Živorad R. Lazić

Design of Experiments in Chemical Engineering

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Živorad R. Lazić

Design of Experiments in Chemical Engineering

A Practical Guide

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Contents

Preface *IX*

I **Introduction to Statistics for Engineers** *1*
1.1 The Simplest Discrete and Continuous Distributions *7*
1.1.1 Discrete Distributions *10*
1.1.2 Continuous Distribution *13*
1.1.3 Normal Distributions *16*
1.2 Statistical Inference *22*
1.2.1 Statistical Hypotheses *23*
1.3 Statistical Estimation *30*
1.3.1 Point Estimates *31*
1.3.2 Interval Estimates *33*
1.3.3 Control Charts *42*
1.3.4 Control of Type II error-β *44*
1.3.5 Sequential Tests *46*
1.4 Tests and Estimates on Statistical Variance *52*
1.5 Analysis of Variance *63*
1.6 Regression analysis *120*
1.6.1 Simple Linear Regression *121*
1.6.2 Multiple Regression *136*
1.6.3 Polynomial Regression *140*
1.6.4 Nonlinear Regression *144*
1.7 Correlation Analysis *146*
1.7.1 Correlation in Linear Regression *148*
1.7.2 Correlation in Multiple Linear Regression *152*

II **Design and Analysis of Experiments** *157*
2.0 Introduction to Design of Experiments (DOE) *157*
2.1 Preliminary Examination of Subject of Research *166*
2.1.1 Defining Research Problem *166*
2.1.2 Selection of the Responses *170*
2.1.3 Selection of Factors, Levels and Basic Level *185*
2.1.4 Measuring Errors of Factors and Responses *191*

Design of Experiments in Chemical Engineering. Živorad R. Lazić
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ISBN: 3-527-31142-4

2.2 Screening Experiments *196*
2.2.1 Preliminary Ranking of the Factors *196*
2.2.2 Active Screening Experiment-Method of Random Balance *203*
2.2.3 Active Screening Experiment Plackett-Burman Designs *225*
2.2.3 Completely Randomized Block Design *227*
2.2.4 Latin Squares *238*
2.2.5 Graeco-Latin Square *247*
2.2.6 Youdens Squares *252*
2.3 Basic Experiment-Mathematical Modeling *262*
2.3.1 Full Factorial Experiments and Fractional Factorial Experiments *267*
2.3.2 Second-order Rotatable Design (Box-Wilson Design) *323*
2.3.3 Orthogonal Second-order Design (Box-Benken Design) *349*
2.3.4 D-optimality, B_k-designs and Hartleys Second-order Designs *363*
2.3.5 Conclusion after Obtaining Second-order Model *366*
2.4 Statistical Analysis *367*
2.4.1 Determination of Experimental Error *367*
2.4.2 Significance of the Regression Coefficients *374*
2.4.3 Lack of Fit of Regression Models *377*
2.5 Experimental Optimization of Research Subject *385*
2.5.1 Problem of Optimization *385*
2.5.2 Gradient Optimization Methods *386*
2.5.3 Nongradient Methods of Optimization *414*
2.5.4 Simplex Sum Rotatable Design *431*
2.6 Canonical Analysis of the Response surface *438*
2.7 Examples of Complex Optimizations *443*

III Mixture Design "Composition-Property" *465*
3.1 Screening Design "Composition-Property" *465*
3.1.1 Simplex Lattice Screening Designs *469*
3.1.2 Extreme Vertices Screening Designs *473*
3.2 Simplex Lattice Design *481*
3.3 Scheffe Simplex Lattice Design *484*
3.4 Simplex Centroid Design *502*
3.5 Extreme Vertices Designs *506*
3.6 D-optimal Designs *521*
3.7 Draper-Lawrence Design *529*
3.8 Factorial Experiments with Mixture *540*
3.9 Full Factorial Combined with Mixture Design-Crossed Design *543*

Appendix *567*
A.1 Answers to Selected Problems *567*
A.2 Tables of Statistical Functions *589*

Index *607*

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Preface

The last twenty years of the last millennium are characterized by complex automatization of industrial plants. Complex automatization of industrial plants means a switch to factories, automatons, robots and self adaptive optimization systems. The mentioned processes can be intensified by introducing mathematical methods into all physical and chemical processes. By being acquainted with the mathematical model of a process it is possible to control it, maintain it at an optimal level, provide maximal yield of the product, and obtain the product at a minimal cost. Statistical methods in mathematical modeling of a process should not be opposed to traditional theoretical methods of complete theoretical studies of a phenomenon. The higher the theoretical level of knowledge the more efficient is the application of statistical methods like design of experiment (DOE).

To design an experiment means to choose the optimal experiment design to be used simultaneously for varying all the analyzed factors. By designing an experiment one gets more precise data and more complete information on a studied phenomenon with a minimal number of experiments and the lowest possible material costs. The development of statistical methods for data analysis, combined with development of computers, has revolutionized the research and development work in all domains of human activities.

Due to the fact that statistical methods are abstract and insufficiently known to all researchers, the first chapter offers the basics of statistical analysis with actual examples, physical interpretations and solutions to problems. Basic probability distributions with statistical estimations and with testings of null hypotheses are demonstrated. A detailed analysis of variance (ANOVA) has been done for screening of factors according to the significances of their effects on system responses. For statistical modeling of significant factors by linear and nonlinear regressions a sufficient time has been dedicated to regression analysis.

Introduction to design of experiments (DOE) offers an original comparison between so-called classical experimental design (one factor at a time-OFAT) and statistically designed experiments (DOE). Depending on the research objective and subject, screening experiments (preliminary ranking of the factors, method of random balance, completely randomized block design, Latin squares, Graeco-Latin squares, Youdens squares) then basic experiments (full factorial experiments, fractional fac-

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torial experiments) and designs of second order (rotatable, D-optimality, orthogonal, B-designs, Hartleys designs) have been analyzed.

For studies with objectives of reaching optima, of particular importance are the chapters dealing with experimental attaining of an optimum by the gradient method of steepest ascent and the nongradient simplex method. In the optimum zone up to the response surface, i.e. response function, one can reach it by applying second-order designs. By elaborating results of second-order design one can obtain square regression models the analysis of which is shown in the chapter on canonical analysis of the response surface.

The third section of the book has been dedicated to studies in the mixture design field. The methodology of approaching studies has been kept in this field too. One begins with screening experiments (simplex lattice screening designs, extreme vertices designs of mixture experiments as screening designs) through simplex lattice design, Scheffe's simplex lattice design, simplex centroid design, extreme vertices design, D-optimal design, Draper-Lawrence design, full factorial mixture design, and one ends with factorial designs of process factors that are combined with mixture design so-called "crossed" designs.

The significance of mixture design for developing new materials should be particularly stressed. The book is meant for all experts who are engaged in research, development and process control.

Apart from theoretical bases, the book contains a large number of practical examples and problems with solutions. This book has come into being as a product of many years of research activities in the Military Technical Institute in Belgrade. The author is especially pleased to offer his gratitude to Prof. Dragoljub V. Vuković, Ph.D., Branislav Djukić, M.Sc. and Paratha Sarathy, B.Sc. For technical editing of the manuscript I express my special gratitude to Predrag Jovanić, Ph.D., Drago Jauković, B.Sc., Vesna Lazarević, B.Sc., Stevan Raković, machine technician, Dušanka Glavač, chemical technician and Ljiljana Borkovic.

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Morristown, February 2004 Živorad Lazić

I
Introduction to Statistics for Engineers

Natural processes and phenomena are conditioned by interaction of various factors. By dealing with studies of cause-factor and phenomenon-response relationships, science to varying degrees, has succeeded in penetrating into the essence of phenomena and processes. Exact sciences can, by the quality of their knowledge, be ranked into three levels. The top level is the one where all factors that are part of an observed phenomenon are known, as well as the natural law as the model by which they interact and thus realize the observed phenomenon. The relationship of all factors in natural-law phenomenon is given by a formula-mathematical model. To give an example, the following generally known natural laws can be cited:

$$E = \frac{mw^2}{2} \quad ; \quad F = ma \quad ; \quad S = vt \quad ; \quad U = IR \quad ; \quad Q = FW$$

The second group, i.e. at a slightly lower level, is the one where all factors that are part of an observed phenomenon are known, but we know or are only partly aware of their interrelationships, i.e. influences. This is usually the case when we are faced with a complex phenomenon consisting of numerous factors. Sometimes we can link these factors as a system of simultaneous differential equations but with no solutions to them. As an example we can cite the *Navier-Stokes'* simultaneous system of differential equations, used to define the flow of an ideal fluid:

$$\begin{cases} \rho \dfrac{DW_X}{\partial X} = -\dfrac{\partial p}{\partial x} + \mu \left[\nabla^2 W_X + \dfrac{1}{3} \dfrac{\partial Q_f}{\partial X} \right] \\ \rho \dfrac{DW_y}{\partial y} = -\dfrac{\partial p}{\partial y} + \mu \left[\nabla^2 W_y + \dfrac{1}{3} \dfrac{\partial Q_f}{\partial y} \right] \\ \rho \dfrac{DW_z}{\partial z} = -\dfrac{\partial p}{\partial z} + \mu \left[\nabla^2 W_z + \dfrac{1}{3} \dfrac{\partial Q_f}{\partial z} \right] \end{cases}$$

An an even lower level of knowledge of a phenomenon is the case when only a certain number of factors that are part of a phenomenon are known to us, i.e. there exists a large number of factors and we are not certain of having noticed all the variables. At this level we do not know the natural law, i.e. the mathematical model by which these factors act. In this case we use experiment (empirical research) in order to reach the noticed natural law.

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As an example of this level of knowledge about a phenomenon we can cite the following empirical dependencies *Darcy-Weisbah's* law on drop of fluid pressure when flowing through a pipe [1]:

$$\Delta p = \lambda \frac{L}{D} \rho \frac{W^2}{2}$$

Ergun's equation on drop of fluid pressure when flowing through a bed of solid particles [1]:

$$\frac{\Delta p}{H} = 150 \left(\frac{1-\varepsilon}{\varepsilon^3} \right)^2 \frac{\mu f}{d_p^2} W + 1.75 \frac{1-\varepsilon}{\varepsilon^2} \rho f \frac{W^2}{d_p}$$

The equation defining warming up or cooling of fluid flows inside or outside a pipe without phase changes [1]:

$$\frac{\alpha}{cG} \left[\frac{c\mu}{\lambda} \right]^{0.67} \left[\frac{LH}{D} \right]^{0.33} \left[\frac{\mu ST}{\mu} \right]^{0.14} = 1.86 / \left(\frac{DG}{\mu} \right)^{0.67}$$

The first case is quite clear: it represents deterministic and functional laws, while the second and third levels are examples of stochastic phenomena defined by stochastic dependencies. Stochastic dependency, i.e. natural law is not expressed in individual cases but it shows its functional connection only when these cases are observed as a mass. Stochastic dependency, thus, contains two concepts: the function discovered in a mass of cases as an average, and smaller or greater deviations of individual cases from that relationship.

The lowest level in observing a phenomenon is when we are faced with a totally new phenomenon where both factors and the law of changes are unknown to us, i.e. outcomes-responses of the observed phenomenon are random values for us. This randomness is objectively a consequence of the lack of ability to simultaneously observe all relations and influences of all factors on system responses. Through its development science continually discovers new connections, relationships and factors, which brings about shifting up the limits between randomness and lawfulness.

Based on the mentioned analysis one can conclude that stochastic processes are phenomena that are neither completely random not strictly determined, i.e. random and deterministic phenomena are the left and right limits of stochastic phenomena. In order to find stochastic relationships the present-day engineering practice uses, apart from others, experiment and statistical calculation of obtained results.

Statistics, the science of description and interpretation of numerical data, began in its most rudimentary form in the census and taxation of ancient Egypt and Babylon. Statistics progressed little beyond this simple tabulation of data until the theoretical developments of the eighteenth and nineteenth centuries. As experimental science developed, the need grew for improved methods of presentation and analysis of numerical data.

The pioneers in mathematical statistics, such as *Bernoulli, Poisson, and Laplace,* had developed statistical and probability theory by the middle of the nineteenth century. Probably the first instance of applied statistics came in the application of probability theory to games of chance. Even today, probability theorists frequently choose

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a coin or a deck of cards as their experimental model. Application of statistics in biology developed in England in the latter half of the nineteenth century. The first important application of statistics in the chemical industry also occurred in a factory in Dublin, Ireland, at the turn of the century. Out of the need to approach solving some technological problems scientifically, several graduate mathematicians from Oxford and Cambridge, including *W. S. Gosset*, were engaged. Having accepted the job in l899, Gosset applied his knowledge in mathematics and chemistry to control the quality of finished products. His method of small samples was later applied in all fields of human activities. He published his method in 1907 under the pseudonym "*Student*", known as such even these days. This method had been applied to a limited level in industry up to 1920. Larger applications were registered during World War Two in military industries. Since then statistics and probability theory are being applied in all fields of engineering.

With the development of electronic computers, statistical methods began to thrive and take an ever more important role in empirical researches and system optimization.

Statistical methods of researching phenomena can be divided into two basic groups. The first one includes methods of recording and processing-description of variables of observed phenomena and belongs to *Descriptive statistics*. As a result of applying descriptive statistics we obtain numerical information on observed phenomena, i.e. statistical data that can be presented in tables and graphs. The second group is represented by statistical analysis methods the task of which is to clarify the observed variability by means of classification and correlation indicators of statistic series. This is the field of that *Inferential statistics*, however, cannot be strictly set apart from descriptive statistics.

The subject of statistical researches are the *Population* (*universe, statistical masses, basic universe, completeness*) and *samples* taken from a population. The population must be representative of a collection of a continual chemical process by some features, i.e. properties of the given products. If we are to find a property of a product, we have to take out a sample from a population that, by mathematical statistics theory is usually an infinite gathering of elements-units.

For example, we can take each hundredth sample from a steady process and expose it to chemical analysis or some other treatment in order to establish a certain property (taking a sample from a chemical reactor with the idea of establishing the yield of chemical reaction, taking a sample out of a rocket propellant with the idea of establishing mechanical properties such as tensile strength, elongation at break, etc.). After taking out a sample and obtaining its properties we can apply descriptive statistics to characterize the sample. However, if we wish to draw conclusions about the population from the sample, we must use methods of statistical inference.

What can we infer about the population from our sample? Obviously the sample must be a representative selection of values taken from the population or else we can infer nothing. Hence, we must select a random sample.

A random sample is a collection of values selected from a population of values in such a way that each value in the population had an equal chance of being selected

Often the underlying population is completely hypothetical. Suppose we make five runs of a new chemical reaction in a batch reactor at constant conditions, and

then analyze the product. Our sample is the data from the five runs; but where is the population? We can postulate a hypothetical population of "all runs made at these conditions now and in the future". We take a sample and conclude that it will be representative of a population consisting of possible future runs, so the population may well be infinite.

If our inferences about the population are to be valid, we must make certain that future operating conditions are identical with those of the sample.

For a sample to be representative of the population, it must contain data over the whole range of values of the measured variables. We cannot extrapolate conclusions to other ranges of variables. A single value computed from a series of observations (sample) is called a "*statistic*".

Mean, median and mode as measures of location

By sample mean $\overline{X}$ we understand the value that is the arithmetic average of property values $X_1, X_2, X_3, , X_i$. When we say average, we are frequently referring to the sample mean, which is defined as the sum of all the values in the sample divided by the number of values in the sample. A sample mean-average is the simplest and most important of all data measures of location.

$$\overline{X} = \sum \frac{X_i}{n} \tag{1.1}$$

where:
$\overline{X}$ is the sample mean-average of the n-values,
X_i is any given value from the sample.

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The symbol $\overline{X}$ is the symbol used for the sample mean. It is an estimate of the value of the mean of the underlying population, which is designated μ. We can never determine μ exactly from the sample, except in the trivial case where the sample includes the entire population but we can quite closely estimate it based on sample mean. Another average that is frequently used for measures of location is the *median*. The median is defined as that observation from the sample that has the same number of observations below it as above it. Median is defined as the central observation of a sample where values are in the array by sizes.

A third measure of location is the *mode*, which is defined as that value of the measured variable for which there are the most observations. Mode is the most probable value of a discrete random variable, while for a continual random variable it is the random variable value where the probability density function reaches its maximum. Practically speaking, it is the value of the measured response, i.e. the property that is the most frequent in the sample. The mean is the most widely used, particularly in statistical analysis. The median is occasionally more appropriate than the mean as a measure of location. The mode is rarely used. For symmetrical distributions, such as the Normal distribution, the mentioned values are identical.

Example 1.1 [2]

As an example of the differences among the three measures of location, let us consider the salary levels in a small company. The annual salaries are:

President	50.000
Salesman	15.000
Accountant	8.000
Foreman	7.000
Two technicians, each	6.000
Four workmen, each	4.000

If the given salaries are put in the array we get:

$$\underbrace{4.000; 4.000; 4.000; 4.000}_{mode};\ \underbrace{6.000}_{median};\ 6.000; 7.000; 8.000; 15.000; 50.000$$

During salary negotiations with the company union, the president states that the average salary among the 10 employees is 9000$/yr, and there is certainly no need for a raise. The union representative states that there is a great need for a raise because over half of the employees are earning 6000$ or less, and that more men are making 4000$ than anything else. Clearly, the president has used the mean; and the union, the median and mode.

Measures of variability, the range, the mean deviation and variance

As we can see, mean or average, median and mode are measure of Location. Having determined the location of our data, we might next ask how the data are spread out about mean. The simplest measure of variability is *range* or *interval*. The range is defined as the difference between the largest and smallest values in the sample.

$$(\text{interval-range}) = X_{max} - X_{min} \tag{1.2}$$

This measure can be calculated easily but it offers only an approximate measure of variability of data as it is influenced only by the limit values of observed property that can be quite different from other values. For a more precise measure of variability we have to include all property-response values, i.e. from all their deviations from the sample mean, mostly the average. As the mean of the values of deviation from the sample mean is equal to null, we can take as measures of variability the *mean deviation*. The mean deviation is defined as the mean of the absolute values of deviation from the sample mean:

$$m = \frac{1}{N}\sum_{i=1}^{N}\left|X_i - \overline{X}\right| \tag{1.3}$$

The most popular method of reporting variability is the *sample variance*, defined as:

$$S_X^2 = \frac{\sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2}{n-1} \tag{1.4}$$

A useful calculation formula is:

$$S_X^2 = \frac{n\sum X_i^2 - (\sum X_i)^2}{n(n-1)} \tag{1.5}$$

The sample variance is essentially the sum of the squares of the deviation of the data points from the mean value divided by (n-1). A large value of variance indicates that the data are widely spread about the mean. In contrast, if all values for the data points were nearly the same, the sample variance would be very small. The *standard deviation* s_x is defined as the square root of the variance. The standard deviation is expressed by the same units as random variable values. Both standard deviation and the average are expressed by the same units. This characteristic made it possible to mutually compare variability of different distributions by introducing the relative measure of variability, called the *coefficient of variation:*

$$k_v = \frac{S_X}{\overline{X}} = \frac{S_X}{\overline{X}} 100\% \tag{1.6}$$

A large value of variation coefficient indicates that the data are widely spread about the mean. In contrast, if all values for the data points were nearly the same, the variation coefficient would be very small.

Example 1.2 [2]
Suppose we took ten different sets of five random observations on X and then calculated sample means and variances for each of the ten groups.

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Sample	Value	Sample mean	Sample variance
1	1;0;4;8;0	2.6	11.8
2	2;2;3;6;8	4.2	7.2
3	2;4;1;3;0	2.0	2.5
4	4;2;1;6;7	4.0	6.5
5	3;7;5;7;0	4.4	8.8
6	7;7;9;2;1	5.2	12.2
7	9;9;5;6;2	6.2	8.7
8	9;6;0;3;1	3.8	13.7
9	8;9;5;7;9	7.6	2.8
10	8;5;4;7;5	5.8	2.7
Means		4.58	7.69

We would have ten different values of sample variance. It can be shown that these values would have a mean value nearly equal to the population variance σ_X^2. Similarly, the mean of the sample means will be nearly equal to the population mean μ. Strictly speaking, our ten groups will not give us exact values for σ_X^2 and μ. To obtain these, we would have to take an infinite number of groups, and hence our sample would include the entire infinite population, which is defined in statistics as *Glivenko's* theorem [3].

To illustrate the difference between values of sample estimates and population parameters, consider the ten groups of five numbers each as shown in the table. The sample means and sample standard deviations have been calculated from appropriate formulas and tabulated. Usually we could calculate no more than that these values are estimates of the population parameters μ and σ_X^2, respectively. However in this case, the numbers in the table were selected from a table of random numbers ranging from 0 to 9 – Table A. In such a table of random numbers, even of infinite size, the proportion of each number is equal to 1/10. This equal proportion permits us to evaluate the population parameters exactly:

$$\mu = \frac{0+1+2+3+4+5+6+7+8+9}{10} = 4.50 \quad ;$$

$$\sigma_X^2 = \frac{(0-4.5)^2+(1-4.5)^2+\ldots(9-4.5)^2}{10} = 8.25$$

We can now see that our sample means in the ten groups scatter around the population mean. The mean of the ten group-means is 4.58, which is close to the population mean. The two would be identical if we had an infinite number of groups. Similarly, the sample variances scatter around the population variance, and their mean of 7.69 is close to the population variance.

What we have done in the table is to take ten random samples from the infinite population of numbers from 0 to 9. In this case, we know the population parameters so that we can get an idea of the accuracy of our sample estimates.

Problem 1.1
From the table of random numbers take 20 different sample data with 10 random numbers. Determine the sample mean and sample variance for each sample. Calculate the average of obtained "statistics" and compare them to population parameters.

1.1 The Simplest Discrete and Continuous Distributions

In analyzing an engineering problem, we frequently set up a mathematical model that we believe will describe the system accurately. Such a model may be based on past experience, on intuition, or on a theory of the physical behavior of the system.

Once the mathematical model is established, data are taken to verify or reject it. For example, the perfect gas law (PV = nRT) is a mathematical model that has been found to describe the behavior of a few real gases at moderate conditions. It is a "law" that is frequently violated because our intuitive picture of the system is too simple.

In many engineering problems, the physical mechanism of the system is too complex and not sufficiently understood to permit the formulation of even an approximately accurate model, such as the perfect gas law. However, when such complex systems are in question, it is recommended to use statistical models that to a greater or lesser, but always well-known accuracy, describe the behavior of a system.

In this chapter, we will consider probability theory, which provides the simple statistical models needed to describe the drawing of samples from a population, i.e. simple probability models are useful in describing the presumed population underlying a random sample of data. Among the most important concepts of probability theory is the notion of random variable. As realization of each random event can be numerically characterized, the various values, which take those numbers as definite probabilities, are called random variables. A random variable is often defined as a function that to each elementary event assigns a number. Thus, influenced by random circumstances a random variable can take various numerical values. One cannot tell in advance which of those values the random variable will take, for its values differ with different experiments, but one can in advance know all the values it can take. To characterize a random variable completely one should know not only what values it can take but also how frequently, i.e. what the probability is of taking those values. The number of different values a random variable takes in a given experiment can be final. If random variable takes a finite number of values with corresponding probabilities it is called a *discrete random variable*. The number of defective products that are produced during a working day, the number of heads one gets when tossing two coins, etc., are the discrete random variables. The random variable is *continuous* if, with corresponding probability, it can take any numerical value in a definite range. Examples of continuous random variables: waiting time for a bus, time between emission of particles in radioactive decay, etc.

The simplest probability model

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Probability theory was originally developed to predict outcomes of games of chance. Hence we might start with the simplest game of chance: a single coin. We intuitively conclude that the chance of the coin coming up heads or tails is equally possible. That is, we assign a probability of 0.5 to either event. Generally the probabilities of all possible events are chosen to total 1.0.

If we toss two coins, we note that the fall of each coin is independent of the other. The probability of either coin landing heads is thus still 0.5. The probability of both coins falling heads is the product of the probabilities of the single events, since the single events are independent:

$$P(\text{both heads}) = 0.50 \times 5 = 0.25$$

Similarly, the probability of 100 coins all falling heads is extremely small:

$$P(100 \text{ heads}) = 0.5^{100}$$

A single coin is an example of a "*Bernoulli*" distribution. This probability distribution limits values of the random variable to exactly two discrete values, one with probability p, and the other with the probability (1-p). For the coin, the two values are heads p, and tails (1-p), where $p = 0.5$ for a "fair" coin.

The Bernoulli distribution applies wherever there are just two possible outcomes for a single experiment. It applies when a manufactured product is acceptable or defective; when a heater is on or off; when an inspection reveals a defect or does not. The Bernoulli distribution is often represented by 1 and 0 as the two possible out-

comes, where 1 might represent heads or product acceptance and 0 would represent tails or product rejection.

Mean and variance

The tossing of a coin is an experiment whose outcome is a random variable. Intuitively we assume that all coin tosses occur from an underlying population where the probability of heads is exactly 0.5. However, if we toss a coin 100 times, we may get 54 heads and 46 tails. We can never verify our intuitive estimate exactly, although with a large sample we may come very close.

How are the experimental outcomes related to the population mean and variance? A useful concept is that of the "*expected value*". The expected value is the sum of all possible values of the outcome of an experiment, with each value weighted with a probability of obtaining that outcome. The expected value is a weighted average.

The "mean" of the population underlying a random variable X is defined as the expected value of X:

$$\mu = E(X) = \sum X_i p_i \tag{1.7}$$

where:
μ is the population mean;
E(X) is the expected value of X;

By appropriate manipulation, it is possible to determine the expected value of various functions of X, which is the subject of probability theory. For example, the expected value of X is simply the sum of squares of the values, each weighted by the probability of obtaining the value.

The population variance of the random variable X is defined as the expected value of the square of the difference between a value of X and the mean:

$$\sigma^2 = E(X-\mu)^2 \tag{1.8}$$

$$\sigma^2 = E\left(X^2 - 2X\mu + \mu^2\right) = E\left(X^2\right) - 2\mu E(X) + \mu^2 \tag{1.9}$$

As E(X) = μ, we get:

$$\sigma^2 = E\left(X^2\right) - \mu^2 \tag{1.10}$$

By using the mentioned relations for Bernoulli's distribution we get:

$$E(X) = \sum X_i p_i = (p)(1) + (1-p)(0) = p \tag{1.11}$$

$$E\left(x^2\right) = \sum X_i^2 p_i = (p)\left(1^2\right) + (1-p)\left(0^2\right) = p \tag{1.12}$$

So that μ = p, and σ^2 = p-p^2 for the coin toss:

p = 0.5; μ = 0.5; σ^2 = 0.25;

1.1.1 Discrete Distributions

A discrete distribution function assigns probabilities to several separate outcomes of an experiment. By this law, the total probability equal to number one is distributed to individual random variable values. A random variable is fully defined when its probability distribution is given. The probability distribution of a discrete random variable shows probabilities of obtaining discrete-interrupted random variable values. It is a step function where the probability changes only at discrete values of the random variable. The *Bernoulli* distribution assigns probability to two discrete outcomes (heads or tails; on or off; 1 or 0, etc.). Hence it is a discrete distribution.

Drawing a playing card at random from a deck is another example of an experiment with an underlying discrete distribution, with equal probability (1/52) assigned to each card. For a discrete distribution, the definition of the expected value is:

$$E(X) = \sum X_i p_i \tag{1.13}$$

where:
X_i is the value of an outcome, and
p_i is the probability that the outcome will occur.

The population mean and variance defined here may be related to the sample mean and variance, and are given by the following formulas:

$$E(\overline{X}) = E\left(\sum X_i/n\right) = \sum E(X_i)\Big/n = \sum \mu/n = n\mu/n = \mu \tag{1.14}$$

$$E(\overline{X}) = \mu \tag{1.15}$$

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Equation (1.15) shows that the expected value (or mean) of the sample means is equal to the population mean.

The expected value of the sample variance is found to be the population variance:

$$E\left(S^2\right) = E\left[\frac{\sum \left(X_i - \overline{X}\right)^2}{n-1}\right] \tag{1.16}$$

Since:

$$\sum \left(X_i - \overline{X}\right)^2 = \sum X_i^2 - 2\sum \overline{X} X_i + \sum \overline{X}^2 = \sum X_i^2 - n\overline{X}^2 \tag{1.17}$$

we find that:

$$E\left(S^2\right) = \frac{E\left(\sum X_i^2\right) - nE\left(\bar{X}^2\right)}{n-1} = \frac{\sum EX_i^2 - nE\left(\bar{X}^2\right)}{n-1} \tag{1.18}$$

It can be shown that:

$$E\left(X_i^2\right) = \sigma^2 + \mu^2;\ E(\overline{X})^2 = \frac{\sigma^2}{n} + \mu^2 \tag{1.19}$$

so that:

$$E\left(S^2\right) = \frac{n\left[\sigma^2 + \mu^2\right] - \sigma^2 - n\mu^2}{n-1} \tag{1.20}$$

and finally:

$$E\left(S^2\right) = \sigma^2 \tag{1.21}$$

The definition of sample variance with an (n-1) in the denominator leads to an unbiased estimate of the population variance, as shown above. Sometimes the sample variance is defined as the biased variance:

$$S^2 = \frac{\sum \left(X_i - \overline{X}\right)^2}{n} \tag{1.22}$$

So that in this case:

$$E\left(S^2\right) = \frac{n-1}{n}\sigma^2 \tag{1.23}$$

A more useful and more frequently used distribution is the *binomial* distribution. The binomial distribution is a generalization of the Bernoulli distribution. Suppose we perform a Bernoulli-type experiment a finite number of times. In each trial, there are only two possible outcomes, and the outcome of any trial is independent of the other trials. The binomial distribution gives the probability of k identical outcomes occurring in n trials, where any one of the k outcomes has the probability p of occurring in any one (Bernoulli) trial:

$$P(X = k) = \binom{n}{k} p^k (1-p)^{n-k} \tag{1.24}$$

The symbol n over k is referred to as the combination of n items taken k at a time. It is defined as:

$$\binom{n}{k} = \frac{n!}{k!(n-k)!} \tag{1.25}$$

Example 1.3

Suppose we know that, on the average, 10% of the items produced in a process are defective. What is the probability that we will get two defective items in a sample of ten, selected randomly from the products, drawn randomly from the product population?

Here, n = 10; k = 2; p = 0.1, so that:

$$P(X = 2) = \binom{10}{2} \times (0.1)^2 \times (0.9)^8 = 0.1938$$

The chances are about 19 out of 100 that two out of ten in the sample are defective. On the other hand, chances are only one out of ten billion that all ten would be found defective. Values of P(X=k) for other values may be calculated and plotted to give a graphic representation of the probability distribution Fig. 1.1.

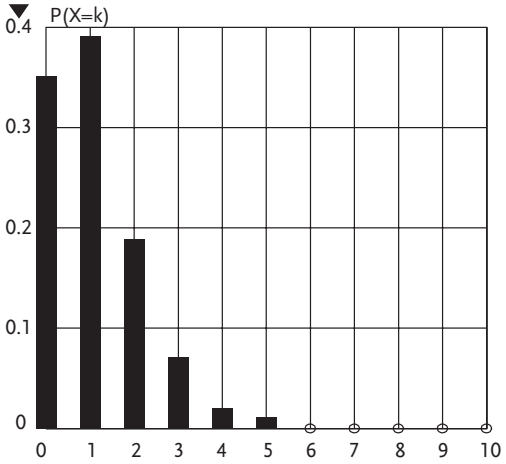

Figure 1.1 Binomial distribution for p = 0.1 and n = 10.

Table 1.1 Discrete distributions.

Distributions	Mean	Variance	Model	Example
Bernoulli $x_i = 1$ with p ; $x_i = 0$ with (1-p)	p	p(1- p)	Single experiment, two possible outcomes	Heads or tails with a coin
Binomial $P_{(x=k)} = \binom{n}{k} p^k (1-k)^{n-k}$	np	np(1- p)	n Bernoulli experiments with k outcomes of one kind	Number of defective items in a sample drawn without replacement from a finite population
Hypergeometric $P_{(X=k)} = \binom{M}{k}\binom{N-M}{n-k} \Big/ \binom{N}{n}$	$\frac{n \times M}{N}$	$\frac{n \times M(N-M)(N-n)}{N^2(N-1)}$	M objects of one kind, N objects of another kind. k objects of kind M found in a drawing of n objects. The n objects are drawn from the population without replacement after each drawing.	Number of defective items in a sample drawn without replacement from a finite population.
Geometric $P_{(X=k)} = p(1-p)^k$	$\frac{1-p}{p}$	$\frac{1-p}{p^2}$	Number of failures before the first success in a sequence of Bernoulli trials.	Number of tails before the first head.
Poisson $P_{(X=k)} = e^{-\lambda t}(\lambda t)^k \Big/ k!$	λt	λt	Random occurrence with time. Probability of k occurrences in interval of width t. λ is a constant parameter	Radioactive decay, equipment breakdown

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One defective item in a sample is shown to be most probable; but this sample proportion occurs less than four times out of ten, even though it is the same as the population proportion. In the previous example, we would expect about one out of ten sampled items to be defective. We have intuitively taken the population proportion ($p = 0.1$) to be the expected value of the proportion in the random sample. This proves to be correct. It can be shown that for the binomial distribution:

$$\mu = np; \sigma^2 = np(1-p) \tag{1.26}$$

Thus for the previous example:

$$\mu = 10 \times 0.1 = 1; \sigma^2 = 10 \times 0.1 \times 0.9 = 0.9$$

Example 1.4 [4]
The probability that a compression ring fitting will fail to seal properly is 0.1. What is the expected number of faulty rings and their variance if we have a sample of 200 rings?

Assuming that we have a binomial distribution, we have:

$$\mu = n \times p = 200 \times 0.1 = 20; \sigma^2 = np \times (1-p) = 200 \times 0.1 \times 0.9 = 18$$

A number of other discrete distributions are listed in *Table 1.1*, along with the model on which each is based. Apart from the mentioned discrete distribution of random variable *hypergeometrical* is also used. The hypergeometric distribution is equivalent to the binomial distribution in sampling from infinite populations. For finite populations, the binomial distribution presumes replacement of an item before another is drawn; whereas the hypergeometric distribution presumes no replacement.

1.1.2 Continuous Distribution

A continuous distribution function assigns probability to a continuous range of values of a random variable. Any single value has zero probability assigned to it. The continuous distribution may be contrasted with the discrete distribution, where probability was assigned to single values of the random variable. Consequently, a continuous random variable cannot be characterized by the values it takes in corresponding probabilities. Therefore in a case of continuous random variable we observe the probability $P(x \le X \le \Delta x)$ that it takes values from the range $(x, x+\Delta x)$, where Δx can be an arbitrarily small number. The deficiency of this probability is that it depends on Δx and has a tendency to zero when $\Delta x \to 0$. In order to overcome this deficiency let us observe the function:

$$f(x) = \lim_{\Delta x \to 0} \frac{P(x \le X \le x+\Delta x)}{\Delta x}; f(x) = \frac{\mathrm{d}P(x)}{\mathrm{d}x} \tag{1.27}$$

which does not depend on Δx and that is called the *probability density function* of continuous random variable X. The probability that the random variable lies between any two specific values in a continuous distribution is:

$$P(a \leq X \leq b) = \int_{a}^{b} f(x)\mathrm{d}x \tag{1.28}$$

where f(x) is the probability density function of the underlying population model. Since all values of X lie between minus infinity and plus infinity $[-\infty; +\infty]$, the probability of finding X within these limits is 1. Hence for all continuous distributions:

$$\int_{-\infty}^{+\infty} f(X)\mathrm{d}x = 1 \tag{1.29}$$

The expected value of a continuous distribution is obtained by integration, in contrast to the summation required for discrete distributions. The expected value of the random variable X is defined as:

$$E(X) = \int_{-\infty}^{+\infty} xf(x)\mathrm{d}x \tag{1.30}$$

The quantity f(x)dx is analogous to the discrete p(x) defined earlier so that Equation (1.30) is analogous to Equation (1.13). Equation (1.30) also defines the mean of a continuous distribution, since μ = E(X). The variance is defined as:

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$$\sigma^2 = \int_{-\infty}^{+\infty} (x-\mu)^2 f(x)\mathrm{d}x \tag{1.31}$$

or by expression:

$$\sigma^2 = \int_{-\infty}^{+\infty} x^2 f(x)\mathrm{d}x - \left[\int_{-\infty}^{+\infty} xf(x)\mathrm{d}x\right]^2 \tag{1.32}$$

The simplest continuous distribution is the *uniform* distribution that assigns a constant density function over a region of values from a to b, and assigns zero probability to all other values of the random variable Figure 1.2.

The probability density function for the uniform distribution is obtained by integrating over all values of x, with f(x) constant between a and b, and zero outside of the region between a and b:

$$\int_{-\infty}^{+\infty} f(x)\mathrm{d}x = \int_{a}^{b} f(x)\mathrm{d}x = 1 \tag{1.33}$$

After integrating this relation, we get:

$$f(x) = \frac{1}{\int_a^b dx} = \frac{1}{b-a}; f(x) = \text{const} \tag{1.34}$$

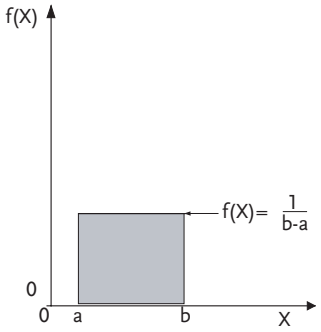

Figure 1.2 Uniform Distribution

Next to follow is:

$$\mu = E(X) = \int_a^b \frac{x dx}{b-a} = \frac{1}{2}(a+b) \tag{1.35}$$

We also get that:

$$\sigma^2 = \int_a^b \frac{x^2 dx}{b-a} - \left[\frac{1}{2}(a+b)\right]^2 = \frac{(b-a)^2}{12} \tag{1.36}$$

Example 1.5

As an example of a uniform distribution, let us consider the chances of catching a city bus knowing only that the buses pass a given corner every 15 min. On the average, how long will we have to wait for the bus? How likely is it that we will have to wait at least 10 min.?

The random variable in this example is the time T until the next bus. Assuming no knowledge of the bus schedule, T is uniformly distributed from 0 to 15 min. Here we are saying that the probabilities of all times until the next bus are equal. Then:

$$f(t) = \frac{1}{15-0} = \frac{1}{15}$$

The average wait is:

$$E(T) = \frac{\int_0^{15} t\, dt}{15} = 7.5$$

Waiting at least 10 min. implies that T lies between 10 and 15, so that by Eq. (1.28):

$$P(10 \leq T \leq 15) = \int_{10}^{15} \frac{dt}{15} = \frac{1}{3}$$

In only one case out of three will we need to wait 10 min. or more. The probability that we will have to wait exactly 10 min. is zero, since no probability is assigned to specific values in continuous distributions. Characteristics of several continuous distributions are given in Table 1.2.

Table 1.2 Continuous distributions

Distribution and density	Mean	Variance	Model	Example
Uniform $f(x) = 1/(b-a)$a<x<b	$\frac{1}{2}(a+b)$	$\frac{(b-a)^2}{12}$	f(x) = constant	Waiting for a bus
Negative exponentials $f(x) = \lambda e^{-\lambda x} \quad ; x > 0$	$\frac{1}{\lambda}$	$\frac{1}{\lambda^2}$	Describes distribution of time between the successive events in a Poisson distribution	Time between emission of particles in radioactive decay.
Normal $f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2\right]$	μ	σ^2	Gaussian distribution	Many experimental situations
Standard normal $f(y) = \frac{1}{\sqrt{2\pi}} \exp\left(-\frac{y^2}{2}\right)$	0	1	A special case of the normal distribution	Many experimental situations
Chi-square $f(x) = \frac{x^{\left(\frac{k}{2}-1\right)} \exp(-\frac{x}{2})}{\left(\frac{k}{2}-1\right)!\, 2^{\frac{k}{2}}} \quad ; x > 0$	k	2k	Distribution of a sum of squares of independent standard normal variables. k is referred to as "degrees of freedom"	Statistical tests on assumed normal distribution.

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1.1.3 Normal Distributions

The normal distribution was proposed by the German mathematician *Gauss*. This distribution is applied when analyzing experimental data and when estimating random errors, and it is known as Gauss' distribution. The most widely used of all continuous distributions is the normal distribution, for the folowing reasons:

- many random variables that appear during an experiment have normal distributions;
- large numbers of random variables have approximately normal distributions;

- if a random variable have not does a normal distribution, not even approximately, it can then be transformed into a normal random variable by relatively simple mathematical transformations;
- certain complex distributions can be approximated by normal distribution (binomial distribution);
- certain random variables that serve for verification of statistical tests have normal distributions.

Gauss assumed that any errors in experimental observations were due to a large number of independent causes, each of which produced a small disturbance. Under this assumption the well-known bell-shaped curve has been obtained. Although it adequately describes many real situations involving experimental observations, there is no reason to assume that actual experimental observations necessarily conform to the Gaussian model. For example, *Maxwell* used a related model in deriving a distribution function for molecular velocities in a gas; but the result is only a very rough approximation of the behavior of real gases. Error in experimental measurement due to combined effects of a large number of small, independent disturbances is the primary assumption involved in the model on which the normal distribution is based. This assumption leads to the experimental form of the normal distribution.

The assumption of a normal distribution is frequently and often indiscriminately made in experimental work, because it is a convenient distribution on which many statistical procedures are based. Many experimental situations, subject to random error, yield data that can be adequately described by the normal distribution, but this is not always the case.

The terms μ and σ^2 are initially defined simply as parameters in the normal distribution function. The term μ determines the value on which the bell-shaped curve is centered and σ^2 determines the "spread" in the curve Fig. 1.3.

A large variance gives a broad, flat curve, while a small variance yields a tall, narrow curve with most probabilities concentrated on values near μ.

The mean or expected value of the normal distribution is obtained by applying Eq. (1.28):

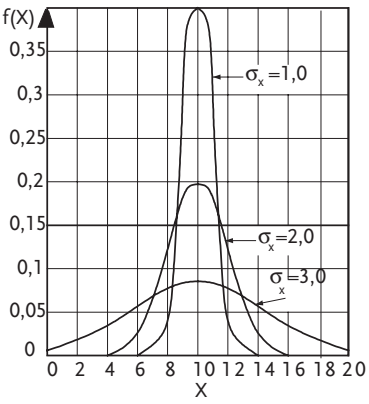

Figure 1.3: How the varaince affects normal distribution curve.

$$E(X) = \frac{1}{\sigma\sqrt{2\pi}} \int_{-\infty}^{+\infty} X \exp\left[-\frac{1}{2}\left(\frac{X-\mu}{\sigma}\right)^2\right] dx \tag{1.37}$$

Integration of Eq. (1.37) is tedious, and is most easily accomplished by using a table of definite integrals. To integrate, we define a new variable:

$$y = \frac{x-\mu}{\sigma} \Rightarrow x = \sigma y + \mu; dx = \sigma dy \tag{1.38}$$

Since μ and σ are constant, Eq. (1.37) becomes:

$$E(X) = \frac{1}{\sigma\sqrt{2\pi}} \left[\int_{-\infty}^{+\infty} \sigma^2 y \exp\left(-\frac{y^2}{2}\right) dy + \mu\sigma \int_{-\infty}^{+\infty} \exp\left(-\frac{y^2}{2}\right) dy\right] \tag{1.39}$$

We find that the first integral is null. For the second integral, we get from a table of definite integrals:

$$\int_{-\infty}^{+\infty} \exp\left(-\frac{y^2}{2}\right) dy = \sqrt{2\pi} \Rightarrow E(X) = \mu \tag{1.40}$$

A similar analysis shows that the variance of X is simply σ_X^2. *The standard normal distribution* is obtained by defining a *standardized variable* z:

$$z = \frac{x-\mu}{\sigma} \Rightarrow x = \sigma z + \mu; dx = \sigma dz \tag{1.41}$$

So that the probability density function becomes:

$$f(z) = \frac{1}{\sqrt{2\pi}} e^{-\frac{z^2}{2}} \tag{1.42}$$

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In this case, $\mu_z = 0; \sigma_z^2 = 1$. The graph of standard normal distribution is illustrated in Fig. 1.4, and a brief tabulation given in Table 1.3 from Table B.

About 68 % of the area under the curve lies between z = 1 and z = +1 (one standard deviation on either side of the mean). Statistically, from Eq. (1.28):

$$P(-1 \le z \le 1) = \int_{-1}^{+1} f(z) dz = \frac{1}{\sqrt{2\pi}} \int_{-1}^{+1} \exp\left(-\frac{z^2}{2}\right) dz$$

The integral is evaluated from Table 1.3:

$$P(-1 \le z \le 1) = P(z \le 1) - P(z \le -1) = 0.8413 - 0.1587 = 0.6816$$

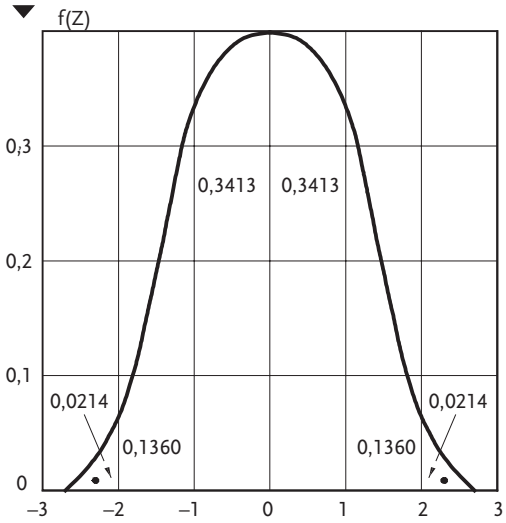
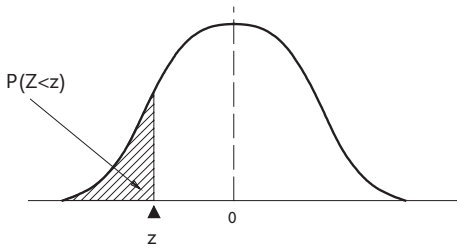


Figure 1.4 Standard normal distribution curve



$$P(Z \leq z) = \int_{-\infty}^{z} \frac{1}{\sqrt{2\pi}} \exp\left(-\frac{y^2}{2}\right) \mathrm{d}y$$

Table 1.3 Abbreviated table of standard normal distribution

z	0.0	0.1	0.2	0.3	0.4	0.5	0.9
–3.0	0.0013						
–2.0	0.0228	0.179	0.0139	0.0107	0.0082	0.0062	0.0019
–1.0	0.1587	0.1357	0.1151	0.0968	0.0808	0.0668	0.0287
–0.0	0.500	0.4602	0.4207	0.3821	0.3446	0.3085	0.1841
+0.0	0.500	0.5398	0.5793	0.6179	0.6554	0.6915	0.8159
+1.0	0.8413	0.8643	0.8849	0.9032	0.9192	0.9332	0.9713
+2.0	0.9772	0.9821	0.9893	0.9893	0.9918	0.9938	0.9981
+3.0	0.9987						

Example 1.6

For example, suppose we have a normal population with $\mu = 6; \sigma^2 = 4$, and we want to know what percentage of the population has values greater than 9?

By Eq. (1.41), $z = \frac{9-6}{2} = 1.5$. From Table 1.3 P(z < 1.5) = 0.9332, and P(z > 1.5) = 1–P(z < 1.5) = 1–0.9332 = 0.0668. Hence about 6.68 % of the population has values greater than 9.

■ **Problem 1.2 4**

Using Table 1.3 i.e. Table B for standard normal distribution, determine probabilities that correspond to the following Z intervals.

a) $0 \le Z \le 1.4$;	b) $-0.78 \le Z \le 0$;	c) $-0.24 \le Z \le 1.9$;
d) $0.75 \le Z \le 1.96$;	e) $-\infty Z \le 0.44$;	f) $-\infty Z \le 1.2$;
g) $-1 \le Z \le 1$;	h) $-1.96 \le Z \le 1.96$;	i) $-2.58 \le Z \le 2.58$

Approximations to discrete distribution

It has already been mentioned that certain distributions can be approximated to a normal one. As the size of the sample increases, the binomial distribution asymptotically approaches a normal distribution. This is a useful approximation for large samples.

Example 1.7

For example, suppose that we wish to know the probability of obtaining 40 or less heads in 100 tosses of a coin. From Eq. (1.24), we sum all 40 values of P:

$$P(X \le 40) = \sum_{k=0}^{40} \binom{100}{k} (0.5)^{100}$$

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This expression would be very tedious to evaluate, so we use the normal approximation. By Eq. (1.26) we get:

$$\mu = 100 \times 0.5 = 50 \; ; \quad \sigma^2 = 100 \times 0.5 \; (1–0.5) = 25$$

Then, by Eq. (1.41) it follows:

$$Z = \frac{40-50}{5} = -2.0; P(z \le -2) = 1 - P(z \le 2) = 1 - 0.9772 = 0.0228$$

so that: P(X<40) = 0.0228 or 2.28 %. For small samples, the approximation is improved by adding 0.5 to the X in Eq. (1.41):

$$Z = \frac{X+0.5-np}{\sqrt{np(1-p)}} \tag{1.43}$$

Problem 1.3

Determine z_1 if the probability is that Z is between:

a) $P(-\infty \leq Z \leq z_1) = 0.92$
b) $P(-1.6 \leq Z \leq z_1) = 0.03$

Problem 1.4

A sample of 36 observations was drawn from a normally distributed population having a mean of 20 and variance of 9. What portion of the population can be expected to have values greater than 26?

Problem 1.5 [4]

The average particulate concentration in micrograms per cubic meter for a station in a petrochemical complex was measured every 6 hours for 30 days. The resulting data are given in table.

5	7	9	12	13	16	17	19	23	24	41
18	24	6	10	16	14	23	19	8	20	26
15	6	11	16	12	22	9	8	15	18	13
7	13	14	8	17	19	11	21	9	55	72
23	24	12	220	25	13	8	9	20	61	48
565	65	10	43	20	45	27	20	72	12	115
130	82	55	26	52	34	66	112	40	34	89
85	95	28	110	16	19	61	67	45	34	32
103	72	67	30	21	122	42	125	50	57	56
25	15	46	30	35	40	16	53	65	78	98
80	65	84	91	71	78	58	26	48	21	

A new air pollution regulation requires that the total particulate concentration be kept below 70+5 mg m^3.

a) What is the probability that the particulate concentration on any day will fall within this allowed range?
b) What is the probability of exceeding the upper limit?
c) What is the probability of operating in the absolutely safe region below 65 mg/m^3?

Problem 1.6:

A random variable has a normal distribution with the following parameters: $\mu = 8; \sigma = 4$. Find:

a) P(5<X<10); b) P(10<X<15); c) P(X>15); d) P(X<5)?

Problem 1.7

Let us suppose that the body weights of 800 students have a normal distribution with mean $\mu = 66$ kg and standard deviation $\sigma = 5$ kg. Find the number of students whose weight is:

a) between 65 and 75 kg;
b) over 72 kg.

■ **Problem 1.8**

A machine is mounted for production of metal rods 24 cm long, with a tolerance rate of $\varepsilon = 0.05$ cm. Based on long-time observation it is established that $\sigma = 0.03$. On the assumption that lengths of X metal rods have a normal distribution calculate the percentage of metal rods that will be placed in tolerance range. How large should this tolerance be so that 95 per cent of produced metal rods should be within these tolerance limits?

■ **Problem 1.9**

In the lab mixer 20 batches of a composite rocket propellant were mixed under identical conditions, all of them having the same composition. Test strands were taken out of the obtained propellant and their burning rates were measured at 70 bar of pressure. The burning rate average is $\bar{X}$=8.5 mm/s , and the calculated variance is σ^2=0.30. What number of strands has the burning rate:

a) between 8 and 9 mm/s;
b) over 9 mm/s;
c) below 8 mm/s.

1.2 Statistical Inference

After gathering a set of experimental data, we usually wish to use it to draw a conclusion about the underlying population. For example, from data on the yield of a chemical reactor, we may want to:

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- Decide whether the average yield from several runs at constant operating conditions equals that required by economic factors;
- Determine whether one set of operating conditions gives a significantly higher yield than another;
- Estimate the average yield to be expected in further runs at specified operating conditions;
- Find a quantitative equation that can be used to predict the yield at various operating conditions.

We can use various methods of statistical inference to arrive at these conclusions. Because the original data are subject to experimental error and may not exactly fit our presumed model, we can draw a conclusion only within specified limits of certainty. We can never make completely unequivocal inferences about a population by using statistical procedures.

Statistical inference may be devided into two broad categories:

- hypothesis testing;
- statistical estimation.

In the first case, we set up a hypothesis about the population and then either accept or reject it by a test using sample data. The first two examples in the first paragraph above involve hypothesis testing. In the first example, the hypothesis would be that the average yield equals the required yield.

Estimation involves the calculation of numerical values for the various population parameters (mean, variance, and so on). These numerical values are only estimates of the actual parameters, but statistical procedures permit us to establish the accuracy of the estimate.

1.2.1 Statistical Hypotheses

A statistical hypothesis is simply a statement concerning the probability distribution of a random variable. Once the hypothesis is stated, statistical procedures are used to test it, so that it may be accepted or rejected. Before the hypothesis is formulated, it is almost always necessary to choose a model that we assume adequately describes the underlying population. The choice of a model requires the specification of the probability distribution of the population parameters of interest to us. When a statistical hypothesis is set up, then the corresponding statistical procedure is used to establish whether the proposed hypothesis should be accepted or rejected. Generally speaking, we are not able to answer the question whether a statistical hypothesis is right or wrong. If the information from the sample taken supports the hypothesis, we do not reject it. However, if those data do not back the statistical hypothesis set up, we reject it.

In principle, two hypotheses are set up:

- primary or null hypothesis H_0;
- alternative hypothesis H_1

If we accept the null hypothesis H_0 we automatically reject the alternative hypothesis H_1.

A large number of statistical hypotheses are of the kind that test specific or range values of one or more distribution parameters. Such hypotheses are tested by using the properties of sample data. As simple drawing of a sample from a population does not have to mean that we obtained a completely representative sample, we are likely apt to make certain errors even when accepting or rejecting a hypothesis.

Types of errors

When testing statistical hypotheses, two types of error may be defined, together with their probability of occurrence.

- *Type I error:* Rejecting H_0 when it is true. Let α equal probability of rejecting H_0 when it is true. This term is also referred to as the "level of significance" of the test.
- *Type II error:* Accepting H_0 when it is false (that is, when H_1 is true). Let β equal the probability of accepting H_0 when it is false.

One can generally say that α and β are risks of accepting false hypotheses. Ideally we would prefer a test that minimized both types of errors. Unfortunately, as α decreases, β tends to increase, and vice versa. Apart from the terms mentioned we should introduce the new term *power of a test*. The power of a test is defined as the probability of rejecting H_0 when it is false. Symbolically it is: *power of a test* = $1-\beta$ or probability of making a correct decision.

The test statistic

To make a test of the hypothesis, sample data are used to calculate a test statistic. Depending upon the value of the test statistic, the primary hypothesis H_0 is accepted or rejected. The critical region is defined as the range of values of the test statistic that requires a rejection H_0. The test statistic is determined by the specific probability distribution and by the parameter selected for testing.

Procedure for testing a hypothesis

The general procedure for testing a statistical hypothesis is:

1. Choose a probability model and a random variable associated with it. This choice may be based on previous experience or intuition.
2. Formulate H_0 and H_1. These must be carefully formulated to permit a meaningful conclusion.
3. Specify the test statistic.
4. Choose a level of significance α for the test.
5. Determine the distribution of the test statistic and the critical region for the test statistic.
6. Calculate the value of the test statistic from a random sample of data.
7. Accept or reject H_0 by comparing the calculated value of the test statistic with the critical region.

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The following examples illustrate the procedure for a statistical test [7]. In the first, we consider a very simple test on a single observation. The second applies the seven-step procedure to a test on the mean of a binomial population using a normal approximation. Here, and in the third example, we introduce the idea of one-sided and two-sided tests, while in the fourth example we illustrate the calculation of Type II error, and the power function of a test.

Example 1.8

A single observation is taken on a population that is believed to be normally distributed with a mean of 10 and a variance of 9. The observation is X=16. Can we conclude that the observation is from the presumed population?

To answer this question, we follow the seven-step procedure:

1. The probability model is a normal distribution: μ=10; σ^2=9. The random variable is the value of X.

2. The primary hypothesis H_0:X=16 , is from a population that is normally distributed with N (10; 9).
   The alternate hypothesis H_1: X=16, is not from the presumed population.
3. Since we have only one observation for X, the test statistic is simply its standardized value, $Z = (X - \mu)/\sigma_X$. The standard normal tables give the distribution of this statistic.
4. Choice of the level of significance α: is arbitrary. We will use α=0.01 and α=0.05 because one of these values is commonly used.
5. The test statistic is distributed normally with μ=10 and σ_X^2=9, if H_0 is true. A value of X that is too far above or below the mean should be rejected, so we select a critical region at each end of the normal distribution. As illustrated in Fig. 1.5, a fraction 0.025 of the total area under the curve is cut off at each end for α=0.05. From the tables (Table B), we determine that the limits corresponding to these areas are Z=-1.96 and Z=1.96; so that if our single observation falls between these values, we accept H_0. The corresponding values for α=0.01 are Z=±2.58.
6. Find the value for:

$$Z = \frac{16-10}{\sqrt{9}} = 2.0$$

7. Since 2>1.96, we reject H_0 at α=0.05. But since 2<2.58, we accept H_0 at α=0.01.

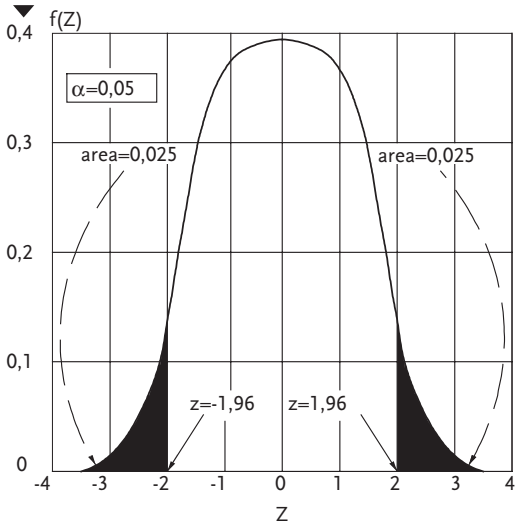

Figure 1.5 Critical region for two-sided test

If we are willing to risk rejecting H_0 when it is true five times out of 100, we can reject it here; but if we wish to reduce the risk of rejecting a true hypothesis to one chance out of 100, then we must accept H_0 for the example. Normally, we do not

carry along two values for α. A single desired level of significance is chosen initially, and the decision is based on it. This almost trivial example illustrates the test procedure and demonstrates the meaning of the level of significance of a test.

Example 1.9

Experience in tire manufacture at a given plant shows that an average of 4.8% of the tires are rejected as imperfect. In a recent day of operation, 60 out of 1000 tires were rejected. Is there any reason to believe that the manufacturing process is functioning improperly?

This problem can be reduced to a test on the sample value. We may use the data on long-range operation to estimate the population parameter ($p=0.048$), since we have no other way of knowing it. We again follow the seven-step method:

1. This is a binomial distribution with two outcomes: acceptance or rejection. However, since the sample size is large, we may use the normal approximation. Since $p=0.048$, we know that the population mean is: $\mu=np=1000 \times 0.048=48$
   and the population variance is:
   $\sigma^2=np\,(1-p)=1000 \times 0.048(1-0.048)=45.7$
2. As the problem is stated, the primary hypothesis is not clearly shown. We choose to compare the mean of the population that is presumed to underlie the day's production to the long-range population mean:

   $H_0:\mu\leq 48; \qquad H_1:\mu>48$

   The question then is whether the day's rejection rate of 60 is sufficiently high to reject the hypothesis that the rejection rate is 48 or less.
3. Since we are using a normal approximation, the test statistic is simply the standard normal variable:

   $$Z=\frac{X-0.5-\mu_0}{\sigma}$$

   The subtraction of 0.5 from the sample value improves the normal approximation. It is called the "continuity correction". The numerator of the equation is the deviation of the sample value from the population mean. The denominator is simply the standard deviation of the presumed population.
   Thus, Z is the number of standard deviations away from the mean at which we find the sample value X. Here we have used the sample value X as an estimate of the population mean presumed to underlie the day's production.
4. Let $\alpha=0.05$. That is, we will risk rejecting the true hypothesis that $\mu=48$ is one chance in 20.
5. To determine the critical region, we must know the distribution of the test statistic. In this case, Z is distributed as the standard normal distribution.
   With $H_0:\mu<48$ and $\alpha=0.05$, we determine that the critical region will include 5% of the area on the high end of the standard normal curve Fig. 1.6. The Z-value that cuts off 5% of the curve is found to be 1.645, from a table of

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normal distribution such as Table B. Therefore, if our calculated Z is greater then 1.645 we reject H_0.

6. For this case, σ^2=45.7; X=60; μ=48; n=1000, and:

$$Z = \frac{60-0.5-48}{\sqrt{45.7}} = 1.70$$

7. Since 1.70 >1.645, we reject H_0 and calculate that a rejection rate of 60 out of 1000 tires is significantly higher than the 4.8 % rejection rate. We might now proceed to seek the cause of this change by checking the manufacturing process.

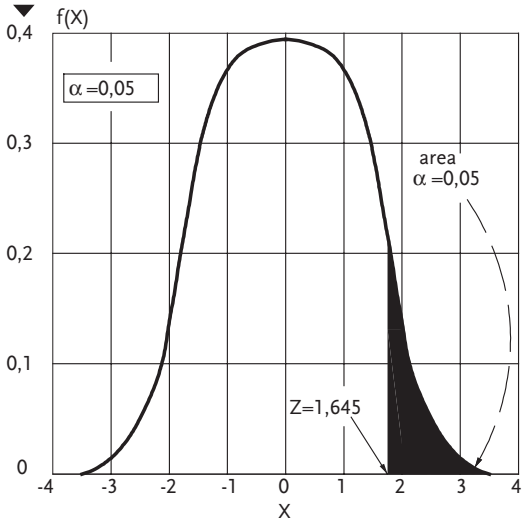

Figure 1.6 Critical region for one-sided test

Suppose instead of finding 60 imperfect tires in 1000, we had found 6 in a sample of 100. Then our solution gives:

$$Z = \frac{6-0.5-4.8}{\sqrt{4.57}} = 0.327; \quad \alpha = 0.05$$

Therefore, with α= 0.05 as before, we would accept H_0. Rejection of 6 tires out of 100 is not significantly different from the population proportion of 0.048, but 60 out of 1000 is significantly different. This illustrates the effect of sample size on statistical tests. A smaller sample is more influenced by random fluctuations than is a larger one; so that the same proportionate difference in a larger sample is statistically more significant than in a smaller one.

This example illustrates a *one-sided* test, that is the critical region is on one side of the probability distribution because of the way the hypotheses are stated.

Example 1.10

Suppose we had chosen to test whether the daily rejection rate of 60 out of 1000 was significantly different from the population proportion of 0.048, rather than significantly higher than the population proportion as in the previous example. In this case, the hypotheses would be:

H_0: μ=48; H_1: μ<48 or μ>48

This is *two-sided* test. The critical region is split into two parts, one rejecting values that are too low and the other rejecting values that are too high. With $\alpha = 0.05$, each critical region has an area of $\alpha/2$=0.25. As shown in Example 1.8 Fig. 1.5, the corresponding Z-values are Z=±1.96. We will reject H_0 if the calculated Z is less than -1.96 or greater than 1.96. With a two-sided test, H_0 is accepted for 60/1000 and 6/100. For this problem, we would probably be more concerned with a rejection rate that was too high, so that the one-sided test would be more appropriate than the two-sided one.

Example 1.11

Determine the power of the test in Example 1.10 for the alternate hypothesis that the mean is really 50, using the sample size 1000.

In this case, we choose a single value from the alternate hypothesis of Example 1.10. From H_1 : μ>48, we select H_1: μ=50.

The power varies with the value selected for the alternate hypothesis. Our hypotheses are then:

H_0: μ=48 ; H_1: μ=50

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The two distributions based on these hypotheses are plotted in Fig. 1.7, where both variances are presumed to be 45.7 (Example 1.9).

With $\alpha = 0.05$, for the two-sided test, Z=1.96 and $X = 48 + 1.96\sqrt{45.7} = 61.3$.

This is the lower limit of the upper critical region as shown under the H_0 curve Fig. 1.7. Similarly, $X = 48 - 1.96\sqrt{45.7} = 34.7$ is the upper limit of the lower critical region. Therefore if X lies between 34.7 and 61.3, H_0 is accepted regardless of whether it is true or not.

Here we have omitted the continuity correction. This is permissible for large sample sizes. In addition, the method used here is only approximate because it assumes the variance is constant regardless of H_1. If H_1:μ=50 were true and H_0 thereby false, the curve for H_1 in Fig. 1.7 would be the correct one; but the limits 34.7 and 6l,3 still define the region of acceptance of H_0. Because H_0 would be accepted when H_1 is true if X falls between (34.7; 61.3), then the area under the curve H_1 between these limits is β, which is the probability of a type II error. The area is labeled in Fig. 1.7. To determine this area, we use the original limits with the curve for H_1 to determine the standardized limits of the β region.

$$\text{Upper limit}: Z = \frac{61.3-50}{\sqrt{45.7}} = 1.67$$

$$\text{Lower limit}: Z = \frac{34.7-50}{\sqrt{45.7}} = -2.26$$

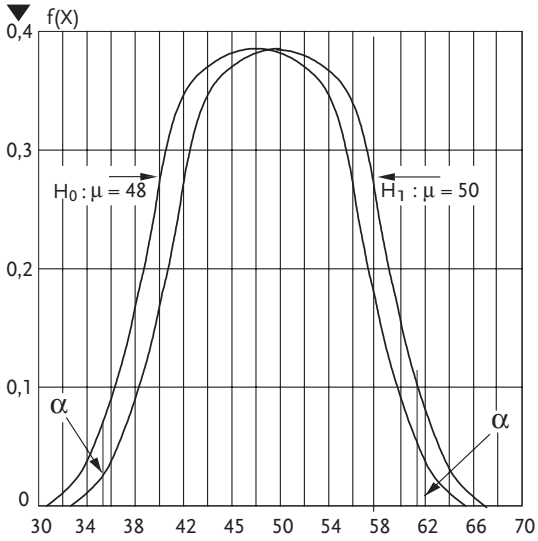

Figure 1.7 Evaluation of type II error

From the normal tables, Table B we have:

$P(Z \leq 1.67)=0.9525$; $P(Z \leq -2.26)=1-P(Z \leq 2.26)=1-0.9881=0.0119$,

Therefore:

$\beta=P(-2.26 \leq Z \leq 1.67)=0.9525-0.0119=0.9406$.

The power is therefore only:

$1-\beta=0.0594$.

Suppose now that we repeat the calculation of the power for other specific values of H_1 and plot them as shown in Fig. 1.8. Inspection of Fig. 1.8 shows that the power would vary from a value of α where $H_1:\mu=48$ to a value of 1.0 where $H_1:\pm\infty$. Such calculations yield the power function curve, as shown in Fig. 1.8. As expected, the further μ_1 is removed from $\mu_0=48$, the higher is the probability of rejecting the false hypothesis H_0. Inspection of Fig. 1.7 shows that β decreases as α increases, so that we could obtain a higher power at the sacrifice of the level of significance. A higher power at the same α is possible if a large sample size is used.

In this part, we have considered the fundamentals of statistical tests and have seen that no test is free from possible error. We can reduce the probability of rejecting a true hypothesis only by running a greater risk of accepting a false hypothesis. We note that a larger sample size reduces the probability of error.

A demand for a specially high level of significance of at least 0.9999 is present in rocket technology and spacecraft industry. In order to reach the mentioned level of significance, there has to be an almost disappearing level of β so that there is no chance of mounting a defective part into the mentioned crafts.

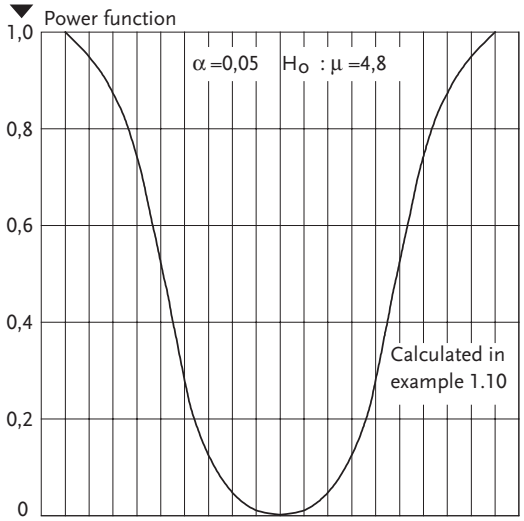

Figure 1.8 Power function for Example 1.10

1.3 Statistical Estimation

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Engineers are often faced with the problem of using a set of data to calculate quantities that they hope will describe the behavior of the process from which the data were taken. Because the measured process variable may be subject to random fluctuations as well as to random errors of measurement, the engineers calculated estimate is subject to error, but how much? Here is where the method of *statistical estimation* can help.

Statistical estimation uses sample data to obtain the best possible estimate of population parameters. The p value of the Binomial distribution, the μ value in Poison's distribution, or the μ and σ values in the normal distribution are called parameters. Accordingly, to stress it once again, the part of mathematical statistics dealing with parameter distribution estimate of the probabilities of population, based on sample statistics, is called *estimation theory*. In addition, estimation furnishes a quantitative measure of the probable error involved in the estimate. As a result, the engineer not only has made the best use of this data, but he has a numerical estimate of the accuracy of these results.

Estimates of two kinds can be made, *point estimate* and *interval estimate*.

Point estimate uses the sample data to calculate a *single* best value, which estimates a population parameter. The point estimate is one number, a point on a numeric axis, calculated from the sample and serving as approximation of the unknown population distribution parameter value from which the sample was taken. Such a point estimate alone gives no idea of the error involved in the estimation. If parameter estimates are expressed in ranges then they are called *interval estimates*.

J. Neuman calls these intervals *confidence intervals*, for as parameter-interval estimates, ranges with known confidence level are chosen. An interval estimate gives a range of values that can be expected to include the correct value with a certain specified percentage of the time. This provides a measure of the error involved in the estimate. The wider the range of the interval estimate, the poorer the point estimate.

1.3.1 Point Estimates

The best point estimate depends upon the criteria by which we judge the estimate. Statistics provides many possible ways to estimate a given population parameter, and several properties of estimates have been defined to help us choose which is best for our purposes.

Example 1.12 [8]
Suppose that we have made 11 runs on a pilot-plant reactor at constant conditions and have obtained the following values of the percentage yield of desired product: 32, 55, 58, 59, 59, 60, 63, 63, 63, 63, 67.

The data fluctuate because of uncontrolled variables and measurement error. Suppose we want the best single value of the yield, where "best" means the yield that we can expect in future runs at the same conditions. We could calculate the sample mean $\bar{X}$=58.4, the median m=60 and the mode 63. But perhaps the 32% yield was a run involving some error of which we are unaware. We cannot arbitrarily drop the run without knowing the cause of the low value, but the mean places undue weight on it.

We might use the median (60) as the best estimate of future yields, since the median does not weight the lowest value unduly. On the other hand, we were able to obtain 63% yield 4 times out of 11. Perhaps this value (the mode) is the best estimate of future operation at carefully controlled conditions. Obviously, statistics cannot make the judgments required in this example. If we can say that the sample mean (58.4) is the best estimate of the population under certain conditions, providing the data come from a random sample of the population. If the 32% yield is as likely to occur as any other value, then the mean is the best estimate.

Several properties of estimates help us to determine which estimate is best for our purposes. We will consider three here:

- consistency
- bias,
- efficiency.

Consistency
This tends to give the correct value of the population parameter as the sample size is increased. For example, as n approaches infinity, the sample mean $\bar{X}$ tends to the population mean μ so $\bar{X}$ is a consistent estimate of μ.

Bias

An estimate is unbiased if on the average it predicts the correct value. Mathematically, an estimate is unbiased if its expected value is equal to the population parameter that it is estimating. For example, the sample mean $\bar{X}$ is an unbiased estimate of the population mean μ because:

$$E(\bar{X})=\mu$$

On the other hand, the sample variance defined by Eq. (1.4) is biased:

$$S_X^2 = \frac{\sum_i (X_i-\bar{X})^2}{n}$$

Because, as shown:

$$E\left(S_X^2\right) = \frac{n-1}{n}\sigma_X^2$$

This means that the condition has not been fulfilled:

$$E\left(S_X^2\right) = \sigma_X^2$$

or that this kind of variance estimation is not unbiased but biased. The population variance unbias estimation is given by the sample variance in the following form:

$$S_X^2 = \frac{\sum_{i=1}^{n} (X_i-\bar{X})^2}{n-1}$$

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Random variable estimations have, apart from the mean, their own variance. It has been proved that when choosing an estimation it is not sufficient to require an estimation to be consistent and biased. It is easy to cite examples of different estimations for consistent and biased basic population means. The criterion for a better estimation is: an estimation is better the smaller dispersion it has. Let us assume that we have two consistent and biased estimations θ_1 and θ_2 for a population parameter and let us suppose that θ_1 has smaller dispersion than θ_2. Fig. 1.9 presents distributions of the given estimations.

It is clear from the figure that both estimations have the same mean, or:

$$E(\theta_1)=E(\theta_2)=\text{ population parameter}$$

The random variable values θ_1 are more centered around the population parameter than the θ_2 ones (i.e. estimations). This means that the average error made in multiple population parameter estimation by means of θ_1 will be smaller than when we do the same for θ_2. The θ_1 estimation can be said to be more efficient.

Efficiency

This is an important property of an estimate. An efficient estimate is the one that gives values that are in general closest to the correct value. A measure of the spread of values around a true value is the variance. The statistical estimate that has the smallest variance is said to be the efficient estimate, and all others are compared

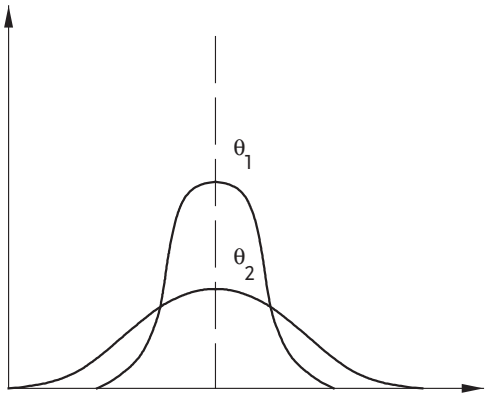

Figure 1.9 Probability distributions of estimations θ_1 and θ_2

with it by taking the ratio of the variances. For example, for a normally distributed population, the mean has the minimum variance σ_X^2 / n; while the variance of the median is $\pi\sigma_X^2 / 2n$. The efficiency of the median as an estimate of the location of the normal population is then:

$$E = \frac{\sigma_X^2 / n}{\pi\sigma_X^2 / 2n} = \frac{2}{\pi} = 0.637 \qquad \text{or} \qquad 63.7\%$$

Efficiency is usually a more important property than unbiasedness. A statistic may prove to be unbiased because large deviations on either side of the correct value cancel each other, but it would be highly inefficient.

1.3.2 Interval Estimates

As it has been mentioned, apart from point estimates there exist the parameter interval estimates. No matter how well the parameter estimate has been chosen, it is only logical to test the estimate deviation from its correct value, as obtained from the sample. For example, if in numerical analysis one obtains that the solution of an equation is approximately 3.24 and that ±0.03 is the maximal possible deviation from the unknown correct solution of the equation, then we are absolutely sure that the range (3.24-0.03=3.21; 3.24+0.03=3.27) contains the unknown correct solution of the equation. Therefore the problem of determining the interval estimate is formulated in the following way:

Let the observed property of X elements of a population have a distribution determined by density function f(X). Let us randomly draw from this population a sample of n observations $X_1, X_2, \ldots, X_n$. We determine two values for the beforehand probability (1-α) close to 1.0 , so that the unknown population parameter value is within this range, while the α probability is outside it. Interval limits are determined by the given sample $X_1, X_2, \ldots, X_n$. We say that $(Z_{\alpha/2}; Z_{1-\alpha/2})$ is the *confidence interval* for the population parameter if its correct value is within the range with beforehand given probability 1-α.

The probability 1-α is called the *confidence coefficient* of the confidence interval. The interval defined in this way is referred to as a *confidence interval*, and the ends of the interval are called *confidence limits*. The quantity (1-α) is the *confidence coefficient*, we can write down:

$$P\left(Z_{\alpha/2} \leq population\ \ parameter \leq Z_{1-\alpha/2}\right) = 1-\alpha \tag{1.44}$$

The interval $(Z_{\alpha/2};Z_{1-\alpha/2})$ is a random variable changed from sample to sample. Some of these intervals will contain the population parameter, others not. However in a large sample, the relative frequency of cases when the interval will contain the population parameter will be approximately 1-α. For a case when it does not have the population parameter, the relative frequency does not go over α.

If we, for instance, choose that 1-α=0.95 we can expect about 95 % of samples to give the confidence interval containing the population parameter. These 0.95 intervals will be called 95 % confidence intervals.

By choosing 1-α=0.99, we can expect the confidence interval to contain the population parameter in some 99 out of 100 cases. But, as will be shown later, the confidence interval corresponding to the coefficient 1(α=0.99, is greater than the one in the case 1-α=0.95. This increase in confidence interval is the bad outcome of the confidence coefficient increase. Which of the 1-α confidence coefficient values to choose in the actual case depends on what error risk is acceptable.

Confidence interval for the mean the variance of which is known

Let us suppose that from a normal population the value μ of which is unknown, the variance σ_X^2 known, a sample $X_1,X_2,...,X_n$. was drawn. The confidence interval for μ should be determined.

Based on *central limit theorem* [3] the average $\bar{X}$ has a normal distribution $n\left(\mu, \sigma_X^2/n\right)$, or:

$$E\left(\bar{X}\right) = \mu\ ;\ \sigma_{\bar{X}}^2 = \sigma_X^2/n \tag{1.45}$$

For testing the hypothesis on the population mean we can use the following statistic:

$$Z = \frac{\bar{X}-\mu}{\sigma_X/\sqrt{n}} \tag{1.46}$$

For a two-sided test, we noted that when Z was less than $z_{\alpha/2}$ or greater than $z_{1-\alpha/2}$ we rejected the primary hypothesis H_0. We accepted H_0 if Z lay between $z_{\alpha/2}$ and $z_{1-\alpha/2}$. The probability that Z lies between these limits is therefore simply (1-α). Stating this mathematically, we get:

$$P(z_{\alpha/2}<Z<z_{1-\alpha/2})=1-\alpha \tag{1.47}$$

Substituting for Z gives:

$$P(z_{\alpha/2}<\frac{\bar{X}-\mu}{\sigma_X/\sqrt{n}}<z_{1-\alpha/2})=1-\alpha \tag{1.48}$$

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Or, the confidence interval is:

$$z_{\alpha/2}<\frac{\bar{X}-\mu}{\sigma_X/\sqrt{n}}<z_{1-\alpha/2} \Rightarrow z_{\alpha/2}\frac{\sigma_X}{\sqrt{n}}<\bar{X}-\mu<z_{1-\alpha/2}\frac{\sigma_X}{\sqrt{n}} \tag{1.49}$$

Then we reverse the signs and add $\bar{X}$ to each term to give:

$$\bar{X}-z_{1-\alpha/2}\frac{\sigma_X}{\sqrt{n}}<\mu<\bar{X}-z_{\alpha/2}\frac{\sigma_X}{\sqrt{n}} \tag{1.50}$$

Since $z_{\alpha/2}=-z_{1-\alpha/2}$ we may rewrite Eq. (1.50) in a more conventional form:

$$\bar{X}+z_{\alpha/2}\frac{\sigma_X}{\sqrt{n}}<\mu<\bar{X}+z_{1-\alpha/2}\frac{\sigma_X}{\sqrt{n}} \tag{1.51}$$

Substituting Eq. (1.51) in Eq. (1.48) then gives:

$$P\left(\bar{X}+z_{\alpha/2}\frac{\sigma_X}{\sqrt{n}}<\mu<\bar{X}+z_{1-\alpha/2}\frac{\sigma_X}{\sqrt{n}}\right)=1-\alpha \tag{1.52}$$

Equation (1.52) gives an interval estimate of μ. The estimate is centered on $\bar{X}$ and extends ($z_{1-\alpha/2}\sigma_X/\sqrt{n}$) on either side of it.

Example 1.13

From the reactor data given earlier, 32, 55, 58, 59, 59, 60, 63, 63, 63, 63, 67 determine an interval estimate of yield, presuming that the population variance is $\sigma_X^2=81$.

Earlier we found the sample mean to be $\bar{X}=58.4$. For $\alpha=0.05$; $Z_{\alpha/2}=-1.96$ and $Z_{1-\alpha/2}=+1.96$. Substituting the values into Eq. (1.52) then gives:

$$P\left(58.40-1.96\frac{9}{\sqrt{11}}\prec\mu\prec 58.40+1.96\frac{9}{\sqrt{11}}\right)=0.95 \tag{1.53}$$

$$P(53.09\prec\mu\prec 63.71)=0.95 \tag{1.54}$$

The interval defined in this way is referred to as a confidence interval, and the ends of the interval are called confidence limits. The quantity (1-α) is the confidence coefficient. We must remember that $\bar{X}$ and hence the confidence limits are the random variables in this statistic; whereas μ is a constant. Thus, with continued sampling, we could obtain other sets of confidence limits. For example, suppose we make another set of 11 runs and get yields of:

47, 53, 56, 58, 58, 61, 61, 62, 64, 64, 65,

Then,

$\bar{X}=59.0$,

and the confidence interval is:

$$P(63.69\prec\mu\prec 64.31)=0.95 \tag{1.55}$$

Equation (1.54) states that 95 out of 100 of these calculated random intervals will contain μ. Or less precisely, we can be 95% confident that the interval calculated actually

contains μ. If we want to be more certain, we must use a smaller level of significance α. For example, with α=0.01, the interval estimate of Eq. (1.54) becomes:

$$P(51.41 \prec \mu \prec 65.39) = 0.99 \tag{1.56}$$

This broader confidence interval is more likely to contain μ. If we want to be absolutely certain (P=1.0) that the interval contains μ, we must write:

$$P(-\infty \prec \mu \prec \infty) = 1.0 \tag{1.57}$$

which gives a rather useless interval. The interval estimated in Eq. (1.54) is sometimes stated another way:

$$\mu=58.40 \pm 5.31(\alpha=0.05) \tag{1.58}$$

Hypothesis testing with interval estimates

The confidence interval may be used to test a hypothesis about the population parameter on which the confidence interval is based. For example, suppose that by using the data of Example 1.13 we wished to test:

$H_0 : \mu=57$

$H_1 : \mu<57 \quad$ or $\quad \mu>57$

If the confidence interval includes the hypothesized value given in H_0, then we accept H_0. For the first set of data, Eq. (1.54) shows that we should accept H_0 because 57 lies in the interval:

$$P(53.09 \prec \mu \prec 63.71) = 0.95.$$

This procedure is really equivalent to our earlier method of hypothesis testing, as an inspection of Eqs. (1.47)–(1.52) shows. In this part and the previous one, we have outlined the principles of statistical tests and estimates. In several examples, we have made tests on the mean, assuming that the population variance is known. This is rarely the case in experimental work. Usually we must use the sample variance, which we can calculate from the data. The resulting test statistic is not distributed normally, as we shall see in the next part of this chapter.

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Tests and estimates on the statistical mean

The mean is perhaps the most important single parameter in many experimental situations because it pin-points the basic location of a population.

In most tests and estimates on the mean, it is assumed that the observations in the sample have been drawn independently from a normal population. This assumption is not as restrictive as it first appears because of the **central limit theorem.**

This theorem, which is of far-reaching importance in statistics, states that:

- The sum of identically distributed independent random variables is normally distributed for large sample size regardless of the probability distribution of the population of the random variable,
- When n tends to be infinite, the random variable distribution X tends to have normal distribution.

The consequence is:

If the random variables $X_1, X_2, \ldots, X_n$ are independent and have the same probability distribution where:

$$E(X_i) = \mu;\ E\left(\sigma_X^2\right) = \sigma_X^2$$

then the random variable probability distribution $\bar{X} = \sum X_i / n$ tends to a normal distribution with parameters μ and $\sigma_X / \sqrt{n}$. As a consequence of the central limit theorem, many test statistics may be assumed to be distributed normally providing that the number of observations n is large, and even though we do not know the underlying distribution. The normal approximation to the binomial distribution that we have used earlier results from the application of the central limit theorem, because the binomial distribution is the sum of independent Bernoulli distributions. How large must n be? Strictly speaking, the normal distribution is approached asymptotically as n approaches infinity. Practically, the size of sample depends on the precision desired.

For a binomial distribution, the normal approximation can be used with good accuracy for sample sizes as low as 8, providing the binomial k is arbitrarily increased by 0.5 in calculating the approximate normal statistic. For values of the parameter p near 0 or 1, a larger sample must be used to obtain an accurate approximation.

One conclusion derived from the central limit theorem is that for large samples the sample mean $\bar{X}$ is normally distributed about the population mean μ with variance $\sigma_{\bar{X}}^2$, even if the population is not normally distributed. This means that we can almost always presume that $\bar{X}$ is normally distributed when we are trying to estimate or make a test on μ, providing we have a large sample.

We have also seen that $\bar{X}$ is an unbiased, efficient, consistent estimate of μ, if the sample is from an underlying normal population. If the underlying population deviates substantially from normality, the mean may not be the efficient estimate; and some other measure of location such as the median may be preferable. We have previously illustrated a simple test on the mean with an underlying normal population of known variance. We shall review this case briefly, applying it to tests between two means, and then proceed to tests where the population variance is unknown.

Tests and estimates with variance known

As discussed earlier, the simplest test on the mean presumes an underlying normal population with known variance, and establishes the hypotheses:

$$H_0 : \mu \leq \mu_0 \qquad \mathrm{H}_0 : \mu > \mu_0 \tag{1.59}$$

where μ_0 is some preselected numerical value. These hypotheses yield a one-sided test, with the critical region at the upper side of the normal distribution. We might also formulate other hypotheses:

$$H_0 : \mu \geq \mu_0 \qquad H_1 : \mu \prec \mu_0 \tag{1.60}$$

or:

$$H_0 : \mu = \mu_0 \qquad H : \mu \prec \mu_0 \text{ or } \mu \succ \mu_0 \tag{1.61}$$

The choice among the three sets depends upon what we wish to test. If we wish to show that μ is higher than μ_0, we use the first test. If we wish to test whether μ is less than μ_0, we use the second set. To show that μ is simply unequal to μ_0, we use the third set with a two-sided critical region. All of these sets use the test statistic:

$$Z = \frac{\bar{X} - \mu}{\sigma_X / \sqrt{n}} \tag{1.62}$$

where Z is distributed normally, with mean equal to zero and variance of one, and where σ_X is known. As shown earlier, the interval estimate that is equivalent to this test on μ is:

$$P(\bar{X} + z_{\alpha/2}\frac{\sigma_X}{\sqrt{n}} < \mu < \bar{X} + z_{1-\alpha/2}\frac{\sigma_X}{\sqrt{n}}) = 1-\alpha$$

This interval estimate is really based on the two-sided test of the third set of hypotheses previously given. Although it is possible to define "one-sided" confidence intervals based on the other two sets of hypotheses (1.59) and (1.60), such one-sided intervals are rarely used. By one-sided, we mean an interval estimate that extends from plus or minus infinity to a single random confidence limit. The one-sided confidence interval may be understood as the range one limit of which is the probability level α and the other one $\pm\infty$.

Comparison of two means: variances known

When the two variances are equal, we may test whether two means are equal by using:

$$H_0 : \mu_1 = \mu_2 \quad H_0 : \mu_1 \prec \mu_2 \; or \; \mu_1 \succ \mu_2 \tag{1.63}$$

with the test statistic:

$$Z = \frac{\bar{X}_1 - \bar{X}_2 - \left(\mu_1 - \mu_2\right)}{\sigma_X \sqrt{1/n_1 + 1/n_2}} \tag{1.64}$$

When H_0 is true, $\mu_1 - \mu_2 = 0$, Eq. (1.64) is simplified. The distribution of Z is normal for underlying normal populations, or for large sample sizes. If the two populations have different variances, the test statistic is:

$$Z = \frac{\bar{X}_1 - \bar{X}_2 - \left(\mu_1 - \mu_2\right)}{\sqrt{\sigma_{X_1}^2 / n_1 + \sigma_{X_2}^2 / n_2}} \tag{1.65}$$

where $\sigma_{X_1}^2 \; and \; \sigma_{X_2}^2$ are the known variances of the two populations.

Tests and estimates with variance unknown

Usually when we have collected some data and wish to use them for tests or estimations, we have no idea of the numerical value of the population variance. As a result, the tests requiring known variance cannot be used. Instead, we calculate the *sample*

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variance and use it in place of the population variance. For example, Eq. (1.62) becomes:

$$T = \frac{\bar{X} - \mu_0}{S_X / \sqrt{n}} \tag{1.66}$$

The new statistic "t" is usually referred to as "*student's" t-distribution* Table C, after *W.S. Gosset*, who first worked out its distribution. For a normal population:

$$T = \frac{\left(\bar{X} - \mu_0\right) \Big/ \left(\sigma_X / \sqrt{n}\right)}{\sqrt{\left(\frac{1}{n-1}\right)\left[\frac{(n-1)\, S_X^2}{\sigma_X^2}\right]}} \tag{1.67}$$

where the numerator is the standard normal variable Z and where $(n-1)S_X^2 \Big/ \sigma_X^2$ has a chi-square distribution. The quantity (n-1) is called the "*degrees of freedom*".

Table 1.4 Selected values for t-distribution

Degrees of freedom (n-1)	$t_{0.90}$	$t_{0.95}$	$t_{0.975}$	$t_{0.99}$	$t_{0.995}$
1	3.08	6.31	12.7	31.8	63.7
2	1.89	2.92	4.30	6.96	9.92
3	1.64	2.35	3.18	4.54	5.84
4	1.53	2.13	2.78	3.75	4.60
5	1.48	2.01	2.57	3.36	4.03
6	1.44	1.94	2.45	3.14	3.71
7	1.42	1.90	2.36	3.00	3.50
8	1.40	1.86	2.31	2.90	3.36
9	1.38	1.83	2.26	2.82	3.25
10	1.37	1.81	2.23	2.76	3.17
15	1.34	1.75	2.13	2.60	2.95
20	1.32	1.72	2.09	2.53	2.84
25	1.32	1.71	2.06	2.48	2.79
30	1.31	1.70	2.04	2.46	2.75
∞	1.28	1.64	1.96	2.33	2.58

Remark: For t values at lower values of ; $t_\alpha = -t_{1-\alpha}$
Hence, $t_{0.05} = -t_{0.95} = -2.35$ for (n - 1)=3

A few values of the t-distribution are given in an accompanying table. We note that t values are considerably higher than corresponding standard normal values for small sample size; but as n increases, the t-distribution asymptotically approaches the standard normal distribution. Even at a sample size as small as 30, the deviation from normality is small, so that it is possible to use the standard normal distribution for sample sizes larger than 30 ($n \geq 30$) and in most cases, for $n < 30$ t-distribution is used. This is equivalent to assuming that S_X^2 is an exact estimate of σ_X^2 at large sample sizes ($n > 30$).

We can also evaluate tests and estimates on the mean with variance unknown. If we calculate S_X^2 from data, the test statistic equivalent to Eq. (1.62) is:

$$T = \frac{\bar{X} - \mu_0}{S_X/\sqrt{n}} \tag{1.68}$$

and the confidence interval is:

$$P\left(\bar{X} + t_{\alpha/2} S_X/\sqrt{n} \prec \mu \prec \bar{X} + t_{1-\alpha/2} S_X/\sqrt{n}\right) = 1 - \alpha \tag{1.69}$$

where T is distributed as Student's t with (n-1) degrees of freedom. The seven-step procedure may be used with this T test, as shown in the following example.

Example 1.14

To test the hypothesis that μ=63, use reactor-yield data:

32; 55; 58; 59; 59; 60; 63; 63; 63; 63; 67.

We first calculate the sample variance as follows:

$$S_X^2 = \frac{\sum (X_i - \bar{X})^2}{n-1} = \frac{n\sum X_i^2 - \left(\sum X_i\right)^2}{n(n-1)} = \frac{11 \times 38340 - 642^2}{11(11-1)} = 87.05 \Rightarrow S_X = 9.33$$

The seven-step procedure follows:

1. The underlying distribution is assumed to be normal;
2. H_0: μ=63; H_1: μ<63 or μ>63
3. The test statistic is: $T = \left(\bar{X} - \mu_0\right) \Big/ \left(S_X/\sqrt{n}\right)$
4. Let α=0.05
5. Test statistic T is distributed as Student's t with n-1=10 degrees of freedom. From Table 1.4, we find $t_{1-0.5/2} = t_{0.975} = 2.23$; $t_{0.05/2} = t_{0.025} = -2.23$ for the two-sided test and α=0.05
6. Since $\bar{X} = 58.4 \Rightarrow T = (58.4 - 63.0)/\left(9.33/\sqrt{11}\right) = -1.64$
7. Accept H_0 since -2.23<-1.64<2.23

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Example 1.15

Make an interval estimate of the yield from the reactor in Example 1.14 for a 95% confidence level.

$$P\left(58.4 - 2.23 \times 9.33/\sqrt{11} \prec \mu \prec 58.4 + 2.23 \times 9.33/\sqrt{11}\right) = 0.95$$

$$P(52.14 \prec \mu \prec 64.66) = 0.95$$

Comparison of means, variances unknown

If we wish to compare two means with population variances unknown, we have two situations. The sample variances may be presumed equal or unequal.

If the two variances are unknown but presumed equal, we calculate a *pooled sample variance*:

$$S_p^2 = \frac{(n_1 - 1) S_1^2 + (n_2 - 1) S_2^2}{n_1 + n_2 - 2} \tag{1.70}$$

Then the test statistic is:

$$T = \frac{\bar{X}_1 - \bar{X}_2}{S_p \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} \tag{1.71}$$

where T has (n_1+n_2(2) degrees of freedom. If the two variances are presumed unequal, we use:

$$T = \frac{\bar{X}_1 - \bar{X}_2}{\sqrt{\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}}} \tag{1.72}$$

where T is distributed with f degrees of freedom, and:

$$f = \frac{\left(S_1^2/n_1 + S_2^2/n_2\right)^2}{\frac{\left(S_1^2/n_1\right)^2}{n_1+1} + \frac{\left(S_2^2/n_2\right)^2}{n_2+1}} - 2 \tag{1.73}$$

The interval estimate for the difference in any two means corresponding to the test in Eq. (1.71) is:

$$P\left[\bar{X}_1 - \bar{X}_2 + t_{\alpha/2} S_p \sqrt{\frac{1}{n_1} + \frac{1}{n_2}} \prec \mu_1 - \mu_2 \prec \bar{X}_1 - \bar{X}_2 + t_{1-\alpha/2} S_p \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}\right] = 1 - \alpha \tag{1.74}$$

If we cannot assume that the two variances S_1^2 *and* S_2^2 are equal, i.e. we cannot calculate the "pooled variance", then (1-α) 100 % confidence interval is based on the following statistic:

$$T = \frac{\bar{X}_1 - \bar{X}_2 - \left(\mu_1 - \mu_2\right)}{\left(S_1^2/n_1 + S_2^2/n_2\right)^{0.5}} \tag{1.75}$$

where the T statistic has an approximate t-distribution with f degrees of freedom:

$$f = \frac{\left(S_1^2/n_1 + S_2^2/n_2\right)^2}{\frac{\left(S_1^2/n_1\right)^2}{n_1-1} + \frac{\left(S_2^2/n_2\right)^2}{n_2-1}} \tag{1.76}$$

so that:

$$P\left[-t_{f,1-\alpha/2} \prec T \prec t_{f,1-\alpha/2}\right] = 1 - \alpha \tag{1.77}$$

$t_{f,1-\alpha/2}$-is obtained from the Table C (Appendix).

By replacements we get (1-α) 100 % confidence interval:

$$P\left[\bar{X}_1 - \bar{X}_2 - t_{f,1-\alpha/2}\sqrt{\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}} \prec \mu_1 - \mu_2 \prec \bar{X}_1 - \bar{X}_2 + t_{f,1-\alpha/2}\sqrt{\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}}\right] = 1 - \alpha \tag{1.78}$$

Example 1.16

Using 95% confidence interval, determine whether the mean nitric acid corrosion rate of metal A is different from that of metal B. The data on 10 runs for each metal under identical conditions are:

A: 40; 42; 42; 43; 46; 47; 47; 48; 49; 50;

B: 39; 41; 41; 44; 45; 45; 46; 47; 48; 48.

For metal A and B we have:

A: $\bar{X}_1 = 45.4$; $S_1^2 = 11.60$;

B: $\bar{X}_2 = 44.4$; $S_2^2 = 9.82$;

The variances are nearly the same, so we shall pool them:

$$S_p^2 = \frac{9\times 11.60 + 9\times 9.82}{18} = 10.71\,;\; S_p = 3.27$$

Substituting in Eq. (1.74), with $t_{0.975} = 2.10\,;\; t_{0.025} = -2.10$ for f=18 degrees of freedom:

$$P\left[(45.4 - 44.4) + (-2.10)\times 3.27\sqrt{\frac{1}{10}+\frac{1}{10}} \prec \mu_1 - \mu_2 \prec (45.4 - 44.4) + 2.10 \times 3.27\sqrt{\frac{1}{10}+\frac{1}{10}}\right] = 0.95$$

$$P\left(-2.7 \prec \mu_1 - \mu_2 \prec 4.07\right) = 0.95$$

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Because the interval includes zero, we may accept the hypothesis H_0 that the means for each metal are not different:

H_0: $H_0 : \mu_1 = \mu_2$ or $H_1 : \mu_1 - \mu_2 = 0$

1.3.3 Control Charts

The concept of a confidence interval may be used to set up a statistical control chart on the mean. Let us consider the reactor from Example 1.14. Suppose we want to use the results of the 11 runs to establish a procedure for operation of the reactor in future runs.

Let us establish a criterion whereby we conclude that the reactor is not in control if the mean of five measurements of yield is more than three standard deviations away from the population mean (as determined by the earlier 11 runs). Then we can establish upper and lower control limits on a control range $\bar{X}\pm 3\sigma$, outside of which we initiate corrective action on the reactor operation.

Example 1.17

Construct a control chart for the reactor from Example 1.14. Use this chart to determine whether the reactor is under statistical control for the following averages of five measurements of yield taken at hourly intervals:

Time:	1	2	3	4	5	6	7	8	9	10
$\bar{X}$, Yield:	55	60	62	52	45	44	43	44	42	43

From the earlier data, we take the sample parameters as our estimate of the population parameters

$\mu=\bar{\bar{X}}=58.4; \qquad \sigma_X=S_X=9.33;$

From Eq. (1.69), the upper control limit is:

$$58.4 + 3 \times 9.33/\sqrt{5} = 70.8$$

and the lower control limit is:

$$58.4 - 3 \times 9.33/\sqrt{5} = 46.0$$

These control limits are plotted on the control chart, along with the hourly yield data as shown in Fig. 1.10.

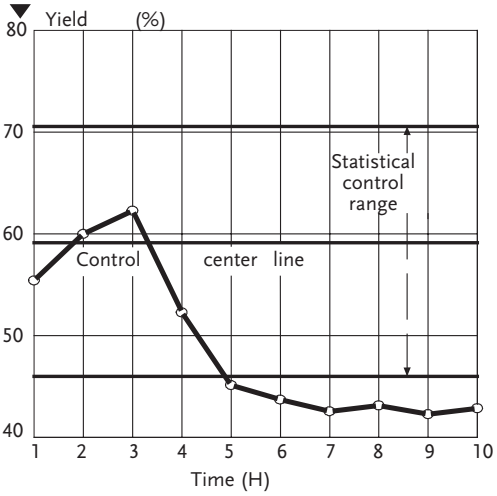

Figure 1.10 Control chart for reactor operation

The reactor was within the control limits until the 5th hour, at which time some corrective action should have been undertaken to restore the yield. As it is, the yield seems to have settled at about 43%, which is outside the control range.

The choice of three standard deviations of the control limits is common. It is equivalent to $\alpha=0.027$. That is, only 27 out of 1000 average yields are likely to fall outside of the control range because of random fluctuations. Therefore we are quite justified in assuming that yields less than 46% are due to something other than random fluctuations.

1.3.4
Control of Type II error-β

Numerous experimental studies and conclusions are brought down to testing the null hypothesis through the obtained observation sample. The so far presented method, i.e. the significance confidence test, takes into account only the type I error α. The new approach, in order to completely fulfill the requirements of an experimenter, chooses the number of sample observations, so that considerably larger differences, useful for the experimenter, can almost always be discovered by a significance test.

This kind of an experiment is said to have type II error control, i.e. the error of not discovering the real deviation from the null hypothesis. The probability of making type II error is marked β.

In experiments in which mean values are compared, the number of observations that should be made depends, as might be expected, on the quantities:
σ-the experimental error standard deviation;
δ-the size of difference between the means it is important to detect;
α-the risk of asserting a difference when none exists; that is the level of probability at which the significance test is made;
β-the risk of asserting no difference when a difference of δ exists.

Obviously, the number of observations of a sample is the function α, β and D=δ/σ. D is the standard difference expressed in standard deviations. To determine the number of observations in a sample for different comparison tests we use Tables F and G.

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When the standard deviation is known, the formula for determing the number of observations in an experiment where two means are compared is easy to be formulate.

The case will be considered in which it is desired to compare the mean $\bar{X}$ of a sample of observations with a standard value μ_0. Suppose $\bar{X}^*$ is the value that the mean of the sample must exceed for the difference to be significant. In accordance with Eq. (1.62) we have:

$$\bar{X}^* = \mu_0 + Z_\alpha \sigma/\sqrt{n} \tag{1.79}$$

If $\bar{X}^*$ is so chosen, there is only a risk α that the sample mean $\bar{X}$ will exceed $\bar{X}^*$ when $\mu = \mu_0$. When μ is equal to $\mu_0 + \delta(\mu - \mu_0 = \delta)$, there exists the risk β of not accepting this assertion but accepting the incorrect hypothesis that $\mu = \mu_0$.

This is the risk that the sample mean $\bar{X}$ falls short of $\bar{X}^*$, and consequently

$$\bar{X}^* = \mu_0 + \delta - Z_\beta \sigma/\sqrt{n} \tag{1.80}$$

Subtracting the second Eq. (1.80) from the first Eq. (1.79) it will be seen that both equations are satisfied when:

$$\delta = \left(Z_\alpha + Z_\beta\right)\sigma/\sqrt{n} \Rightarrow n = \left(Z_\alpha + Z_\beta\right)^2 \sigma^2 \Big/ \delta^2 \tag{1.81}$$

$$n = \left(Z_\alpha + Z_\beta\right)^2 \Big/ D^2 \tag{1.82}$$

where D(=δ/σ) is the difference it is important to detect, expressed as a multiple of the standard deviation

The experimenter should therefore perform the number of experiments indicated by Eq. (1.82) and make a test of significance at the level α.

If the result is significant, we should say that a real difference has occurred; if it is not significant, we should say that no difference as large as δ has occurred. The chances of obtaining a significant result when no difference has occurred and of obtaining a nonsignificant result when a difference $\mu - \mu_0 = \delta$ has occurred will thus be α and β, respectively.

Example 1.18 [9]

Primers of pressed tetryl are used for initiating charges of explosives. A factor affecting their initiating power is their density. For a certain purpose it was desirable that the density of the primers should exceed 1.4 g/cm^3. A scheme was required so that a decision whether to accept the batch as satisfactory or reject it because the average density was too low could be based on the results obtained from testing a fairly small randomly drawn sample of primers from the batch. The standard deviation was known from past experience to be 0.03 and the mean density when the presses were operating normally was 1.54.

A random sample of n primers will be drawn from the batch, the density of each primer measured, and the sample mean is calculated $\bar{X}$. If $\bar{X}$ exceeds the value $\bar{X}^*$, the batch will be accepted; if $\bar{X}$ falls short of this value it will be rejected.

To ensure that good batches are nearly always accepted and bad batches nearly always rejected it was decided that the following requirements should be satisfied:

- If the sample mean density $\bar{X}$was as low as 1.50 there should be a 99% chance of rejection (or a 1% chance of acceptance).
- If the sample mean density assumed the value $\bar{X}$=1.54 there should be a 98% chance of acceptance (or a 2% chance of rejection).

Under the given conditions the value to be compared with the sample mean is 1.50, while the significance level and type II error are α=0.01; β=0.02 and δ=1.54-1.50=0.04, respectively.

Thus: D=δ/σ=0.04/0.03=1.33. The standardized normal distribution table gives: Z_α=1.326 and Z_β=2.054. Using the Eq. (1.82): n=10.8≈11, and the Eq. (1.80): $\bar{X}^*$=1.521. This is illustrated for the present example in Fig. 1.11.

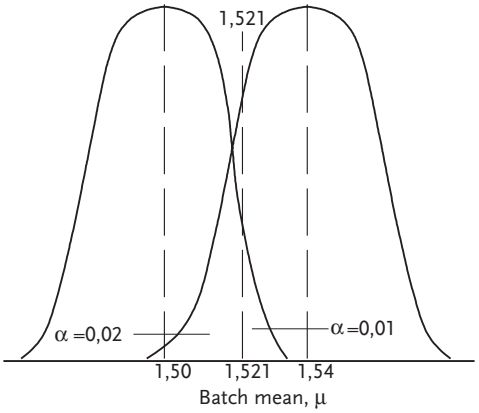

Figure 1.11 Distribution of means for samples of 11 primers

1.3.5
Sequential Tests

The preceding part has indicated the basis on which the experimenter can decide what number of observations is required to make comparative experiments conclusive, on the assumption that this number must be decided before the experiment is performed. When, as is common in chemical and physical research, the observations are obtained one after another, it is generally possible to adopt an alternative procedure in which, after each observation is made, a simple statistical test is applied to determine whether the results obtained so far indicate a definite conclusion from the experiment, or whether more observations are needed to make the experiment decisive. The experiment thus terminates as soon as a definite conclusion can be drawn, and the average number of observations required in experiments carried out in this manner tends to be definitely less than when the number has to be decided beforehand. Consequently this sequential method of performing comparative experiments has definite advantages, particularly when the observations are expensive or time consuming. It is often only half of the number required by non-sequential testing, or in cases of unexpected large effects-differences, sequential testing can offer an answer after only one or two observations. In *sequential testing* a decision is made after each new observation based on all previously obtained information, aimed at asserting whether:

- to accept the null hypothesis that no change of importance has occurred;
- to accept the aslternative hypothesis that a real change has occurred;
- to continue taking observations.

Sequential tests are best explained graphically. As each new observation comes to hand, the value of a function of all observations recorded up to that time is calculated and plotted against the number of observations on a chart such as shown in Fig. 1.12.

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On the chart are two boundary lines, the positions of which depend upon the risks α and β, of errors of type I and type II, the magnitude of the difference it is important to detect, etc. The lines divide the chart into three zones: (1) in which the Null Hypothesis is accepted; (2) in which the alternative hypothesis is accepted; and (3) in which there is no decision.

The sequential test then consists in plotting the function of the observations f(X) against the number of observations n and continuing to take observations so long as the plotted points f(X) fall within (3). As soon as point falls outside this zone, that is, either in zone (1) (acceptance of the H_0), or in zone (2) (acceptance of the H_1), the observations are discontinued and the indicated decision is taken.

In practice the chart is often changed with the boundary values being calculated in advance for each value of n. The test is then made by successive comparisons of the value f(X) with the appropriate limits.

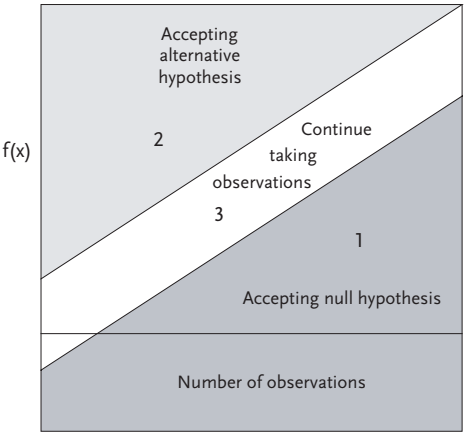

Figure 1.12 Sequential test chart

One-sided sequential testing-comparison of a mean with a standard value

Suppose it is required to test whether the population mean μ of a series of observations is equal to some standard value μ_0. As before, δ denotes the difference it is important to detect, σ the standard deviation, α the risk of asserting a significant difference when none exists, and β the risk of asserting no significant difference when the mean value is really $\mu=\mu_0+\delta$.

The function f(X) plotted for this test is simply the total T of the observations up to the time considered, and the boundaries are parallel straight lines with slope S, cutting the axis of T at h_1 and h_0.. The values h_0, h_1 and S are given by the equations:

$$h_0 = -b\sigma^2 \Big/ \delta;\ h_1 = a\sigma^2 \Big/ \delta;\ S = \mu_0 + 1/2\delta \tag{1.83}$$

$$a = \ln(1-\beta)/\alpha;\ b = \ln(1-\alpha)/\beta \tag{1.84}$$

Table H gives values of a and b for commonly used values of α and β

Example 1.19 [9]

Apply sequential test to Example 1.18 so as to point out the difference between sequential and nonsequential testing. It was assumed that σ was constant and equal to 0.03, and the procedure was planned so that there would only be a small risk (α=0.01) of accepting a bad batch, that is a batch with mean density as low as μ_0=1.50 g/cm^3, and a small risk (β=0.02) of rejecting a good batch, that is a batch with mean density μ_1=1.54 g/cm^3. Thus δ=0.04, σ=0.03, α=0.01, and β=0.02. It was found that for the nonsequential test n=11 observations would be required and that the test should be made by calculating the mean of a sample of eleven, rejecting the batch if the mean was less than $\bar{X}^*$=1.521 g/cm^3 and accepting it otherwise.

Had it been convenient to use a sequential scheme then as each primer was tested the total T of the observations to date would be calculated and plotted on a chart with suitable boundary lines. It will be found in practice that if μ_0 is large compared with δ these boundary lines will rise very steeply and appear to be very close together, so that the chart will be difficult to use. Since the test is to detect a difference, it will not be affected if a constant amount is subtracted from each observation. For purposes of convenience, therefore, instead of considering the actual density we consider the amount by which the density exceeds 1.40, that is to say, 1.40 is subtracted from each observation. Then:

μ_0=0.10; μ_1=0.14; h_0=-0.0878; h_1=0.1032; S=0.12;

To construct the chart convenient values are chosen for the two scales, making the vertical axis the axis of T and the horizontal axis the axis of n. Points are marked off 0.1032 unit above zero and 0.0878 unit below zero on the axis of T, and through these points lines are drawn that rise by 0.12 unit of T for each unit increase in n. A chart is then obtained like that in Fig. 1.13.

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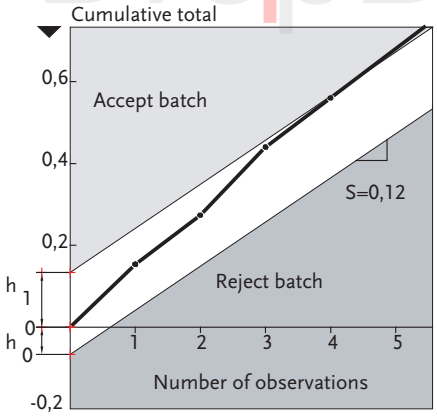

Figure 1.13 Sequential test in control

Suppose, for example, the first five observations were: 1.551; 1.527; 1.581; 1.517; 1.547. Subtracting 1.40, these would be treated as: 0.151; 0.127; 0.181; 0.117; 0.147, and the cumulative totals: 0.151; 0.278; 0.459; 0.576; 0.723, would be plotted against

the value n=1, 2, 3, 4 and 5, respectively. As will be seen from Fig. 1.13, the last point is in the zone of acceptance, and testing would therefore end and the batch would be accepted at this stage.

Alternative methods of presenting the results

In most cases it is quicker to calculate limits T_0 and T_1 for each value of n from the expressions:

$$T_0 = h_0 + ns\,;\; T_1 = h_1 + ns \tag{1.85}$$

Table 1.5 Limit values

n	1	2	3	4	5	6	7	8
T_0	0.0322	0.1522	0.2722	0.3922	0.5122	0.6322	0.7522	0.8722
T_1	0.2232	0.3432	0.4632	0.5832	0.7032	0.8232	0.9430	1.0632

The taking of observations is then continued so long as T lies between T_0 and T_1. In the example given here, assuming as before that 1.40 is subtracted from each observation, these limits are as shown in Table 1.5.

For the particular set of observations previously given, the successive cumulative totals T were: T=0.151; 0.278; 0.459; 0.576 and 0.723. Testing would therefore be discontinued at this stage with acceptance of the batch, since for the first time T falls outside one of the boundaries denoted by T_0 and T_1 (n=5 for T_5=0.723 is outside the range 0.5122–0.7032).

Two-sided sequential testing

The technique so far considered is appropriate for testing whether a mean value is significantly greater than some specified value when the standard deviation is accurately known. A precisely similar procedure is used to test whether a mean value is significantly less than the specified value. When, however, the alternative hypothesis is that μ may depart from μ_0, in either direction, the test procedure will be different. The value δ will now be the deviation (positive or negative) from the specified value that it is desired to detect. A suitable test[1)] can be obtained by superimposing two one-sided tests, say A and B, in each of which the error of the type I is set at $\alpha/2$, and δ is taken to be positive in one test and negative in the other.

The procedure consists of plotting the cumulative sum of the observations, taken with μ_0 as the origin, on a chart such as Fig. 1.14 on which both sets of boundary lines are shown.

The lines divide the chart into a number of zones, which may be merged into three shaded zones and the unshaded zone shown. In the upper shaded zone the hypothesis that a real increase has occurred will be accepted $\mu>\mu_0$, in the center shaded zone the hypothesis that no important change has occurred will be accepted $\mu=\mu_0$, and in the lower shaded zone the hypothesis that a real decrease has occurred will be accepted $\mu<\mu_0$. The logic of this procedure may be seen by considering the

1) This is the two-sided test proposed by Barnard-a

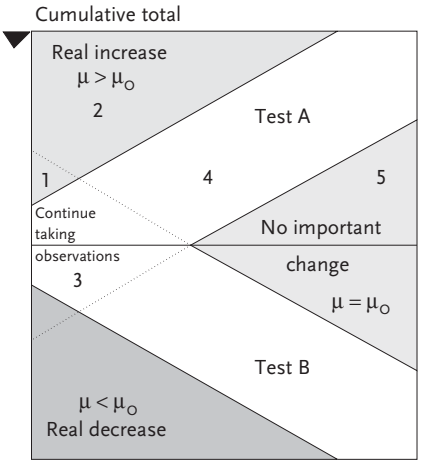

Figure 1.14 Sequential test chart: double-sided testing

nature of the two individual tests A and B. A tests the hypothesis that no increase of importance has occurred $\mu>\mu_0$ against the alternative that a real increase has occurred, and B tests the hypothesis that no decrease of importance has occurred against the alternative that a real decrease has occurred $\mu>\mu_0$. The boundary lines are given by the formulas

$$\left.\begin{aligned} T_0 &= h_0 + ns \\ T_1 &= h_1 + ns \end{aligned}\right\} A \qquad \left.\begin{aligned} T_0 &= h_0 + ns \\ T_1 &= h_1 + ns \end{aligned}\right\} B \tag{1.86}$$

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where:

$$h_0 = -b'\sigma^2 \Big/ \delta = -h_0'$$

$$h_1 = a'\sigma^2 \Big/ \delta = -h_1'$$

$$S = \delta/2 = -S' \tag{1.87}$$

$$a' = \ln(1-\beta)/1/2\alpha$$

$$b' = \ln(1-\alpha/2)/\beta$$

δ is taken to be positive.

The calculations are simplified by the use of Table H, the values of a' and b' being found directly by entering the table with the risk of the error of the type I equal to $1/2\ \alpha$ and the risk of the error of the type II equal to β.

Example 1.20 [9]

In an investigation of factors affecting strength of synthetic fibers a modification was made in the preparation of the material and a series of separate preparations of fiber was made in pairs, one of the regular and one of the modified material; and a number of properties of the resulting fibers were determined. The results for each

pair of observations were known before the next pair were carried out, so that a sequential test could be employed. The most important property measured was the breaking load, and it was known from past experience that the standard deviation of the difference between repeat preparations with respect to this property was approximately 10 units. The experiment was designed so that the risk α of asserting nonexistent changes was α=0.05 and so that a difference of δ=±10 would normally be detected with 90% certainty.

Applying the sequential test to the differences of observed breaking load in repeat pairs of observations, then:

μ_0=0; μ_1=±10 since δ =±10; α=0.05; β=0.1; σ=10; h_0=-22.8; h_1=35.8; S =5.

Based on the mentioned values the boundary lines are drawn:

$$\left.\begin{aligned} T_0 &= -22.8 + 5n \\ T_1 &= 35.8 + 5n \end{aligned}\right\}; \quad \left.\begin{aligned} T_0 &= 22.8 - 5n \\ T_1 &= -35.8 - 5n \end{aligned}\right\};$$

A graphic illustration of a sequential test chart is in Fig. 1.15.

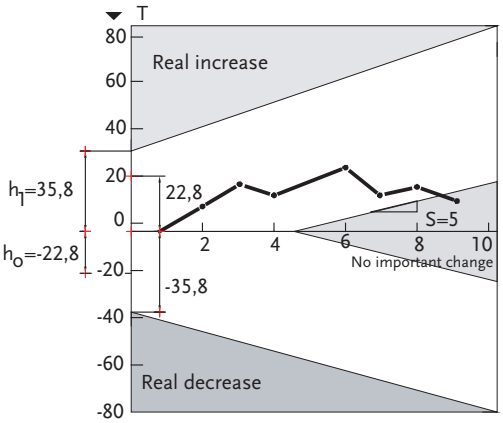

Figure 1.15 Sequential testing-synthetic fiber

In one experiment the following values were recorded for the differences in breaking load between synthetic fibers prepared in two different ways: 7; 5; 8;-11; 10; 8;-9; 6;-7. The cumulative sums of the observations are 7; 12; 20; 9; 19; 27; 18; 24; 17, and the points are plotted in Fig. 1.15. The line crosses the limit T_0 at the ninth observation, so the hypothesis that no important change in breaking load has occurred is accepted. Exactly the same procedure can be done analytically.

1.4
Tests and Estimates on Statistical Variance

After determining the location of a set of data by tests or estimates on the mean, we next check the variability of the data. How seriously do the data scatter about the mean? If the scatter is large, a given observation is less reliable than if the scatter is small.

A measure of the scatter or variability of data is the variance, as discussed earlier. We have seen that a large variance produces broad-interval estimates of the mean. Conversely, a small variability, as indicated by a small value of variance, produces narrow interval estimates of the mean. In the limiting case, when no random fluctuations occur in the data, we obtain exact identical measurements of the mean. In this case, there is no scatter of data and the variance is zero, so that the interval estimate reduces to an exact point estimate.

In practice, random fluctuations in process variables and random errors of measurement are always present. If our measurements are sufficiently sensitive, we will pick up these random fluctuations, and the variance of the measurements will not be zero.

Obviously, we need tests and estimates on the variability of our experimental data. We can develop procedures that parallel the tests and estimates on the mean as presented in the previous section. We might test to determine whether the sample was drawn from a population of a given variance; or we might establish point or interval estimates of the variance. We may wish to compare two variances to determine whether they are equal. Before we proceed with these tests and estimates, we must consider two new probability distributions. Statistical procedures for interval estimates of a variance are based on *chi-square* and *F-distributions*. To be more precise, the interval estimate of a σ^2 variance is based on χ^2-distribution while the estimate and testing of two variances is part of a F-distribution.

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χ^2-chi-square distribution

The chi-square distribution was discussed briefly in the earlier section on probability distributions. Suppose we have (k+1) independent standard normal variables. We then define χ^2 as the sum of the squares of these (k+1) variables. It can be shown that the probability density function of χ^2 is:

$$f\left(x^2\right) = \frac{\left(x^2\right)^{\frac{k}{2}-1} \exp\left(-x^2/2\right)}{\left(\frac{k}{2}-1\right)!\,2^{k/2}} \tag{1.88}$$

Because χ^2 is the square of standard normal variables, it has no negative values:

$$0 \prec \chi^2 \prec \infty$$

The distribution depends on the number of independent variables included in the summation. The parameter of the χ^2 distribution is the degrees of freedom (k),

which is one less than the number of observations. A brief table of the χ^2 distribution is given in Table 1.6, and a few representative curves are shown in the accompanying chart Fig. 1.16:

Table 1.6 Selected values of the χ2-distribution

Degrees of freedom k	$\chi^2_{0.005}$	$\chi^2_{0.01}$	$\chi^2_{0.025}$	$\chi^2_{0.05}$	$\chi^2_{0.95}$	$\chi^2_{0.975}$	$\chi^2_{0.99}$	$\chi^2_{0.995}$
1	0.000	0.000	0.001	0.004	3.84	5.02	6.63	9.88
2	0.010	0.020	0.051	0.103	5.99	7.38	9.21	10.6
3	0.072	0.115	0.216	0.352	7.81	9.35	11.3	12.8
4	0.207	0.297	0.484	0.711	9.49	11.1	13.3	14.9
5	0.412	0.554	0.831	1.15	11.1	12.8	15.1	16.7
6	0.676	0.872	1.24	1.64	12.6	14.4	16.8	18.5
7	0.989	1.24	1.69	2.17	14.1	16.0	18.5	20.3
8	1.34	1.65	2.18	2.73	15.5	17.5	20.1	22.0
9	1.73	2.09	2.70	3.33	16.9	19.0	21.7	23.6
10	2.16	2.56	3.25	3.94	18.3	20.5	23.2	25.2
15	4.60	5.23	6.26	7.26	25.0	27.5	30.6	32.8
20	7.43	8.26	9.59	10.9	31.4	34.2	37.6	40.0
25	10.5	11.5	13.1	14.6	37.7	40.6	44.3	46.9
30	13.8	15.0	16.8	18.5	43.8	47.0	50.9	53.7

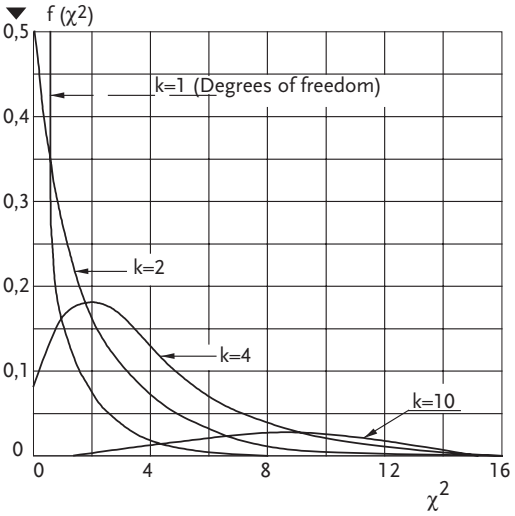

Figure 1.16 Chi-square distributions are unsymmetrical

Based on the value that takes $(0; \infty)$ and Fig. 1.16 we can see that the unsymmetrical distribution is in question. The χ^2-distribution is derived from Maxwell's distribution of molecular velocities in gases [5].

For values k≥30 , the χ^2 -distribution may be approximated from the standard normal distribution:

$$\chi^2_{\alpha,k} = \frac{1}{2}\left[Z_\alpha + \sqrt{2k-1}\right]^2 \tag{1.89}$$

where Z_α is the equivalent percentile of the standard normal variable.

The mean of the χ^2 distribution is k, and the variance is 2k. Because χ^2 is the sum of identically distributed variables, its distribution is asymptotically normal, as shown by the central limit theorem. This can be seen in the accompanying figure for large values of k. For large values of k, we can write:

$$Z = \left(\chi^2 - k\right) \Big/ \sqrt{2k} \tag{1.90}$$

Equation *(1.90)* gives values that are approximately distributed as the standard normal variable. The χ^2 distribution will be used later for tests on the variance, because the following statistic has the χ^2 distribution with k degrees of freedom as shown by *Brownlee* [10].

$$\chi^2_k = k S_X^2 \Big/ \sigma_X^2 \tag{1.91}$$

The F-distribution

The F-distribution is used to compare the variances of two populations. Suppose we calculate the sample variances S_1^2 and S_2^2, for two populations of size n_1 and n_2. Then F is defined as:

$$F = \frac{S_1^2}{S_2^2} \tag{1.92}$$

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where F is distributed as the F-distribution with n_1-1 and n_2-1 degrees of freedom. If we let k_1=n_1-1 and k_2=n_2-1 and use the relations in Eq. (1.91) we may rewrite Eq. (1.92):

$$F = \frac{\sigma_1^2 \chi^2_{k_1} \Big/ k_1}{\sigma_2^2 \chi^2_{k_2} \Big/ k_2} \tag{1.93}$$

If $\sigma_1^2 = \sigma_2^2$, which is frequently the condition being tested, we get:

$$F = \frac{\chi^2_{k_1} \Big/ k_1}{\chi^2_{k_2} \Big/ k_2} \tag{1.94}$$

We see that F is the ratio of two chi-square distributions, each divided by its degrees of freedom. The F-distribution is usually written as $F(k_1, k_2)$, denoting the degrees of freedom. It can be easily shown that:

$$F_\alpha(k_1, k_2) = 1 \,/\, F_{1-\alpha}(k_2, k_1) \tag{1.95}$$

Table 1.7 Selected values of the F-distribution for $F_{\{0.95\}}$

k_2	Degrees of freedom in larger variance, k_1												
	1	2	3	4	5	6	7	8	9	10	20	30	∞
1	161	200	216	225	230	234	237	239	241	242	248	250	254
2	18.51	19.0	19.16	19.25	19.30	19.33	19.36	19.37	19.38	19.39	19.44	19.46	19.50
3	10.13	9.55	9.28	9.12	9.01	8.94	8.88	8.84	8.81	8.78	8.66	8.62	8.53
4	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.80	5.74	5.63
5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.78	4.74	4.56	4.50	4.36
6	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	3.87	3.81	3.67
7	5.59	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.63	3.44	3.38	3.23
8	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.34	3.15	3.08	2.93
9	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.13	2.93	2.86	2.71
10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.97	2.77	2.70	2.54
20	4.35	3.49	3.10	2.87	2.71	2.60	2.52	2.45	2.40	2.35	2.12	2.04	1.84
30	4.17	3.32	2.92	2.69	2.53	2.42	2.34	2.27	2.21	2.16	1.93	1.84	1.62
∞	3.84	2.99	2.60	2.37	2.21	2.09	2.01	1.94	1.88	1.83	1.57	1.46	1.00

Using the Table 1.7, we note that the following value of F gives:

$$F_{0.05}(6;10) = \frac{1}{F_{0.95}(10;6)} = \frac{1}{4.06} = 0.246$$

The F-distribution is very widely used in statistical procedures. It is the distribution used in the analysis of variance, which will be considered later. In this section, we use the F-distribution in tests of equality of the variances of two populations.

Interrelationships of several distributions

In this chapter of statistics for engineers we have so far introduced four important probability distributions used in statistical tests and estimates. These are:

- Standard normal distribution, Z;
- Student's t-distribution, t_k;
- CHI-square distribution, χ^2_k;
- F-distribution, F(k_1, k_2).

Here we summarize several relationships among these four distributions:

1. As the sample size approaches infinity, t approaches the standard normal variable for the same α:

$$t_{k\to\infty} = Z \tag{1.96}$$

This is evident by an inspection of the Tables B and C. For example:

$$t_{0.975\to\infty} = Z_{0.975} = 1.96$$

2. As n →∞ approaches infinity, the ratio χ^2_k/k approaches 1.0. As a result:

$$F_\alpha(k_1, \infty) = \chi^2_{k_1} \Big/ k_1 \tag{1.97}$$

For example, an inspection of Tables 1.6 and 1.7 shows that:

$$F_{0.95}(5, \infty) = \chi^2_{5;0.95} \Big/ 5 = 11.10/5 = 2.21$$

3. The chi-square distribution is related to the normal distribution by:

$$\chi^2_{1,\alpha} = Z^2_{1-\alpha/2} \tag{1.98}$$

For example:

$$\chi^2_{1;0.05} = Z^2_{1-0.05/2} \Rightarrow 3.84 = 1.96^2 = 3.84$$

4. The F-distribution is related to the t-distribution by:

$$F_\alpha(1, k_2) = t^2_{k_2;1-\alpha/2} \tag{1.99}$$

For example:

$$F_{0.95}(1; 10) = t^2_{10;0.975} \Rightarrow 4.96 = 2.23^2 = 4.96$$

Tests and estimates on a single variance

As shown earlier, the sample variance S_X^2 is an ubiased estimate of the population variance σ_X^2. If the sample is from the normal population, S_X^2 is also the efficient estimate; so that S_X^2 is usually the best point estimate of σ_X^2. The mean deviation or range may also be used to estimate the population standard deviation σ_X; but these estimates are biased, less efficient and inconsistent.

If we wish to test whether a sample is drawn from a population of a specific known variance, we have a two-sided test:

$$H_0: \quad \sigma_X^2 = \sigma_0^2$$

$$H_1: \quad \sigma_X^2 \succ \sigma_0^2 \quad \text{or} \quad \sigma_X^2 \prec \sigma_0^2$$

Assuming H_0 is correct, the test statistic is that given in the next form:

$$\chi^2_k = k S_X^2 \Big/ \sigma_0^2 \tag{1.100}$$

The critical region is split between the high and low ends of the distribution:

$$k S_X^2 \Big/ \sigma_0^2 \prec \chi^2_{\alpha/2,k} \quad \text{or} \quad k S_X^2 \Big/ \sigma_0^2 \succ \chi^2_{1-\alpha/2,k} \tag{1.101}$$

If we wish to test whether the variance of a product exceeds a given value, we have a one-sided hypothesis:

$$H_0: \quad \sigma_X^2 \leq \sigma_0^2 \qquad\qquad H_1: \quad \sigma_X^2 \succ \sigma_0^2$$

and a one-sided critical region:

$$k S_X^2 \Big/ \sigma_0^2 \succ \chi^2_{1-\alpha/2,k} \tag{1.102}$$

The confidence interval that is equivalent to the two-sided test is obtained from the critical regions:

$$P\left[k S_X^2 \Big/ \chi^2_{1-\alpha/2,k} \prec \sigma_X^2 \prec k S_X^2 \Big/ \chi^2_{\alpha/2,k}\right] = 1 - \alpha \tag{1.103}$$

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Example 1.21

Determine point and interval estimates for the population variance of the reactor yield data of earlier Example 1.12:

Yield %: 32; 55; 58; 59; 59; 60; 63; 63; 63; 63; 67.

The best point estimate is the sample variance, which was already calculated as S_X^2=87.05 so that S_X=9.33. Using the Eq. (1.103) we get the variance interval estimate for α=0.05:

k=n-1=10; $\chi^2_{0.975} = 20.5;\ \chi^2_{0.025} = 3.25.$

$$P\left(10 \times 87.05/20.5 \prec \sigma_X^2 \prec 10 \times 87.05/3.25\right) = 0.95$$

$$P\left(42.26 \prec \sigma_X^2 \prec 267.85\right) = 0.95$$

The extremly wide range is the consequence of the large sample variance.

Comparison of two variances

The F-statistic may be used to test the quality of two population variances. The hypotheses are:

$$H_0 : \sigma_1^2 = \sigma_2^2 \qquad H_1 : \sigma_1^2 \succ \sigma_2^2 \ \text{ or } \ \sigma_1^2 \prec \sigma_2^2 \tag{1.104}$$

The test statistic is: $F = S_1^2 \Big/ S_2^2$ which has the F-distribution with k_1 and k_2 degrees of freedom.

Example 1.22

Determine whether the assumption of equal variances in Example 1.16 was justified. In the earlier problem, we found:

S_1^2=11.60; n_1=10; S_2^2=9.82; n_2=10.

The seven-step procedure is used:

1. It is necessary to assume that both populations are normally distributed. The random variables are X_1 and X_2, the corrosion rate for each metal A and B.
2. $H_0 : \quad \sigma_1^2 = \sigma_2^2 \qquad H_1 : \sigma_1^2 \succ \sigma_2^2 \ \text{ or } \ \sigma_1^2 \prec \sigma_2^2$
3. The test statistic is: $F = S_1^2 \Big/ S_2^2$
4. Let α=0.10
5. F is distributed as F(9;9), and the critical region is:
   $F \prec F_{0.05}(9;9) \quad and \quad F \succ F_{0.95}\ (9;9)$
   From Table 1.7 or Table E, the value is: $F_{0.95}(9;9)$=3.18;
   $F_{0.05}(9;9)=1/F_{0.95}(9;9)=1/3.18=0.314$
6. For this test: F=11.60/9.82=1.18
7. Since: 0.314<1.18<3.18, we accept H_0. There is no significant difference in the variances.

The interval estimate for the ratio of variances is:

$$P\left[\frac{S_1^2/S_2^2}{F_{1-\alpha/2}\left(k_1,k_2\right)} \prec \frac{\sigma_1^2}{\sigma_2^2} \prec \frac{S_1^2/S_2^2}{F_{\alpha/2}\left(k_1,k_2\right)}\right] = 1-\alpha \tag{1.105}$$

For the previous example:

$$P\left[0.371 \prec \frac{S_1^2}{S_2^2} \prec 3.6\right] = 0.90$$

Recapitulation of statistic tests

Table 1.8 Testing μ when σ^2 is known

Statistic: $Z = \dfrac{\bar{X}-\mu_o}{\sqrt{\sigma^2/n}}$

Null hypothesis	Alternative	Rejection region H_0
$H_0: \mu = \mu_0$	$H_1: \mu \neq \mu_0$	$Z \prec -z_{1-\alpha/2}$ or $Z \succ z_{1-\alpha/2}$
$H_0: \mu \leq \mu_0$	$H_1: \mu > \mu_0$	$Z \succ z_{1-\alpha}$
$H_0: \mu \geq \mu_0$	$H_1: \mu < \mu_0$	$Z \prec -z_{1-\alpha}$

Table 1.9 Testing μ when σ^2 unknown

Statistic: $T = \dfrac{\bar{X}-\mu_o}{S/\sqrt{n}}$

Null hypothesis	Alternative	Rejection region H_0
$H_0: \mu = \mu_0$	$H_1: \mu \neq \mu_0$	$Z \succ t_{n-1,1-\alpha/2}$ or $T \prec -t_{n-1,1-\alpha/2}$
$H_0: \mu \leq \mu_0$	$H_1: \mu \succ \mu_0$	$T \succ t_{n-1,1-\alpha}$
$H_0: \mu \geq \mu_0$	$H_1: \mu \prec \mu_0$	$T \prec -t_{n-1,1-\alpha}$

Table 1.10 Testing μ_1-μ_2 when σ_1^2 and σ_2^2 are known

Statistic: $Z = \dfrac{\bar{X}_1-\bar{X}_2}{\sqrt{\sigma_1^2/n_1+\sigma_2^2/n_2}}$

Null hypothesis	Alternative	Rejection region H_0
$H_0: \mu_1-\mu_2 = 0$	$H_1: \mu_1-\mu_2 \neq 0$	$Z \succ z_{1-\alpha/2}$ or $Z \prec -z_{1-\alpha/2}$
$H_0: \mu_1-\mu_2 \leq 0$	$H_1: \mu_1-\mu_2 \succ 0$	$Z \succ z_{1-\alpha}$
$H_0: \mu_1-\mu_2 \geq 0$	$H_1: \mu_1-\mu_2 \prec 0$	$Z \prec -z_{1-\alpha}$

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Table 1.11 Testing μ_1-μ_2 when σ_1^2 and σ_2^2 unknown but equal

Statistic: $T = \dfrac{\bar{X}_1 - \bar{X}_2}{S_p\sqrt{1/n_1 + 1/n_2}};\ f = n_1 + n_2 - 2$

Null hypothesis	Alternative	Rejection region H_0
$H_0 : \mu_1 - \mu_2 = 0$	$H_1 : \mu_1 - \mu_2 \neq 0$	$T < -t_{f,1-\alpha/2}$ or $T > t_{f,1-\alpha/2}$
$H_0 : \mu_1 - \mu_2 \leq 0$	$H_1 : \mu_1 - \mu_2 \succ 0$	$T > t_{f,1-\alpha}$
$H_0 : \mu_1 - \mu_2 \geq 0$	$H_1 : \mu_1 - \mu_2 \prec 0$	$T = -t_{f,1-\alpha}$

Table 1.12 Testing the variance σ^2

Statistic: $\chi^2 = \dfrac{(n-1)S^2}{\sigma_0^2};\quad k = n - 1$

Null hypothesis	Alternative	Rejection region H_0
$H_0 : \sigma^2 = \sigma_0^2$	$H_1 : \sigma^2 \neq \sigma_0^2$	$\chi^2 \succ \chi^2_{n-1,1-\alpha/2}$ or $\chi^2 \prec \chi^2_{n-1;\alpha/2}$
$H_0 : \sigma^2 \leq \sigma_0^2$	$H_1 : \sigma^2 \succ \sigma_0^2$	$\chi^2 \succ \chi^2_{n-1,1-\alpha}$
$H_0 : \sigma^2 \geq \sigma_0^2$	$H_1 : \sigma^2 \prec \sigma_0^2$	$\chi^2 \prec \chi^2_{n-1,1\alpha}$

Table 1.13 Testing σ_1^2=σ_2^2

Statistic: $F = \dfrac{S_1^2}{S_2^2};\quad k_1 = n_1 - 1;\quad k_2 = n_2 - 1$

Null hypothesis	Alternative	Rejection region H_0
$H_0 : \sigma_1^2 = \sigma_2^2$	$H_1 : \sigma_1^2 \neq \sigma_2^2$	$F \succ F_{k1,k2,1-\alpha/2}$ or $F \prec F_{k1,k2,\alpha/2}$
$H_0 : \sigma_1^2 \leq \sigma_2^2$	$H_1 : \sigma_1^2 \succ \sigma_2^2$	$F \succ F_{k1,k2,1-\alpha}$
$H_0 : \sigma_1^2 \geq \sigma_2^2$	$H_1 : \sigma_1^2 \prec \sigma_2^2$	$F \prec F_{k1,k2,\alpha}$

Problem 1.10 [4]
Successive colorimetric determinations of the normality of a $K_2Cr_2O_7$ solution were as follows 1.22; 1.23; 1.18; 1.31; 1.25; 1.22; 1.24 ($\times 10^{-4}$). Sample variance is $\sigma_{\bar{X}}^2 = 49 \times 10^{-10}$. Determine 95% confidence interval of the mean.

Problem 1.11 [4]
"Frigid-Flow Co." has received final test results on the company's new heat exchanger. The values given below are overall heat-transfer coefficients: 60; 63; 60; 68; 70; 72; 65; 61; 69; 67 BTU/h ft^2 °F. At the 99% confidence level, what minimum value for the exchanger's overall heat-transfer coefficient can the company suggest?

Problem 1.12

Successive determinations of the opened bottles of HCl were found to be, expressed in normalities:

Bottle N1 : 15.75; 15.64; 15.92;
Bottle N2 : 15.58; 15.49; 15.72;

The producer informs that the last delivery has the HCl concentration variance σ^2=0.016. Determine the 95% confidence interval for μ_1-μ_2, where μ_1 and μ_2 are the means of HCl concentrations.

Problem 1.13

In order to compare the effects of two solid catalyst component concentrations on NO_2 reductions, six groups of observations were made. Each group consisted of three replicates of five observations each, that is, a total of 15 determinations were made for each concentration. The concentrations (in mass per cent) were 0.5 and 1.0%. The reduction data are summarized below.

Catalyst type A :	5.18	5.52	5.42
Catalyst type B :	5.58	5.62	5.82.

Do the catalysts have the same efficiency rate?

Problem 1.14 [4]

Five similar determinations of the cold water flow rate to a heat exchanger were, in [GPM]: 5.84; 5.76; 6.03; 5.90; 5.87. Compute a 95% confidence interval for the imprecision affecting this measuring operation?

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Problem 1.15 [4]

Reaction temperatures in degrees centigrade (measured on two different days) for two catalyst concentrations were:

Concentration A: 310.95; 308.86; 312.80; 309.74; 311.03; 311.89; 310.93; 310.39; 310.24; 311.89; 309.65; 311.85; 310.73.

Concentration B: 308.94; 308.23; 309.98; 311.59; 309.46; 311.15; 311.29; 309.16; 310.68; 311.86; 310.98; 312.29; 311.21.

Find a 98% confidence interval for σ_1/σ_2.

Problem 1.16

Data taken from the plate and frame filter press located in the unit operations laboratory are used to determine α, the specific cake resistance, of a calcium carbonate slurry. Several values of α, expressed in ft/lb, have been calculated from data taken during the fall semester.

2.49×10^{11}; 2.40×10^{11}; 2.43×10^{11}; 2.30×10^{11}; 2.53×10^{11}; 2.67×10^{11}; 2.60×10^{11}; 2.50×10^{11}; 2.54×10^{11}; 2.55×10^{11}.

Based on these values, predict the interval within which 90% of all such values calculated in the future must fall.

Problem 1.17

Due to the burning of cotton plant wastes (hulls, leaves, etc.), the sulfate content in the air over a town is highest during the month of November. If the data given below are the mean values of sulfate content during the month of November (analysis of air perform daily) over the past 10 years.

Sulfate content SO_4^{2-} mg/m^3: 10.83; 8.90; 14.71; 12.35; 11.86; 13.80; 11.75; 9.68; 9.33; 10.9.

Determine the 95% confidence interval during next November.

Problem 1.18 [4]

A company engaged in the manufacture of cast iron has employed a system of raw material and processing procedures that has produced a product whose overall population average silicon content was 0.85%. A new contract was put into effect in which a new supplier of raw material supplanted the old one. During the first month of operation using the new material, random samples of the product silicon content were found to be:

1.13; 0.80; 0.85; 0.60; 0.97; 0.92; 0.94; 0.72;
1.17; 0.87; 0.36; 0.68; 0.73; 0.82; 0.79; 0.87;
0.92; 0.81; 0.97; 0.48; 1.00; 0.92; 0.61; 0.81;
0.71; 0.97; 0.89; 0.68; 1.00; 1.16.

What are your 99% confidence limits on the silicon content of the iron using the new raw material.

Problem 1.19 [4]

An experiment conducted to compare the tensile strengths of the types of synthetic fibers gave the breaking strength shown below in thousands of pounds force per square inch (PSI):

Fiber A: 14 4 10 6 3 11 12
Fiber B: 16 17 13 12 7 16 11 8 7.

Calculate the 99% confidence interval for the difference between the means.

Problem 1.20 [4]

The following data were obtained for the calibration of the Ruska dead weight gauge used with our *Burnett PVT Apparatus*. The weights corresponding to the 1.000 PSI loading had the following apparent masses:

26.03570; 26.03581; 26.03529; 26.03573; 26.03575; 26.03551; 26.03588; 26.03586; 26.03599; 26.03533; 26.03570.

Can we say that the average apparent mass does not exceed 26.5?

Problem 1.21

Fifty determinations of a certain concentration yielded the following values:

54.20; 51.73; 52.56; 53.55; 56.15; 57.50; 54.25; 54.46;
53.08; 53.82; 54.15; 53.10; 51.56; 53.43; 53.77; 55.88;
54.96; 58.51; 54.65; 55.13; 51.12; 53.73; 55.01; 55.57;
53.95; 53.39; 54.30; 52.89; 57.35; 55.77; 52.22; 54.55;
56.78; 56.00; 57.27; 54.89; 57.05; 56.25; 56.35; 56.52;
56.91; 52.35; 52.02; 52.94; 58.16; 57.73; 55.33; 54.13;
56.60; 55.21.

Test the hypothesis $H_0 : \mu \geq 55.0$ with the 99% confidence level.

Problem 1.22

Observing the values in Problem 1.12, there is enough reason to believe that both bottles were filled up with HCl in the same production line. The mean variance of concentration for the last year of production is 0.016. Can we trust the assumption that both bottles were filled with HCl from the same batch?

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Problem 1.23

On a pilot-plant for producing composite rocket propellant four batches of the same composition were made under the same process conditions with the batch of 10 micron ammonium perchlorate ground at that moment. A month later the same ammonium perchlorate was used to make three batches of the composite propellants with the same composition. From the obtained propellant experimental rocket motors were static fired. The following burning rates at 70 [bar] pressure were determined from the calculated burning rate laws:

Batch of AP A: 14.199; 14.531; 14.197; 14.193
Batch of AP B: 14.398; 14.418; 14.307.

Test the means of the composite propellant burning rates at 70 [bar].

Problem 1.24

Test the variances in the previous example.

Problem 1.25

On a pilot-plant for producing composite rocket propellant four batches of the same composition were made under the same process conditions with the two different burning-rate catalysts. From the obtained propellant experimental rocket motors were static fired. The following burning rates at 70 [bar] pressure were determined from the calculated burning rate laws:

Catalyst A: 14.199;14.531;14.197;14.193; Catalyst B: 15.716; 15.612; 15.682; 15.715.

Are there significant differences in catalyst effects on burning rates?

1.5 Analysis of Variance

The technique known as *analysis of variance (ANOVA)*[2)] uses tests based on variance ratios to determine whether or not significant differences exist among the means of several groups of observations, where each group follows a normal distribution. The analysis of variance technique extends the t-test used to determine whether or not two means differ to the case where there are three or more means.

The analysis of variance is used very widely in the biological, social and physical sciences. The technique was first developed by *R. A. Fisher* and his colleagues in England in the 1920s. Fisher has said that the analysis of variance is merely *"a convenient way of arranging the arithmetic"*. This statement points out that the statistical principles underlying the analysis of variance are quite simple; but the calculations can become quite involved, so that they require careful and systematic arrangement.

Analysis of variance is particularly useful when the basic difference between the groups cannot be stated quantitatively. For example, suppose we wish to determine whether there are any differences among the effects of four polymerization catalysts on the setting time of a particular plastic. We make several runs under identical conditions with each catalyst. We can then determine whether the mean setting times for the four catalysts are different by using a *one-way analysis of variance* to determine the effect of one independent variable (type of catalyst) on the dependent variable (setting time). However, we cannot describe the type of catalyst by a quantitative relationship. On the other hand, we might run a similar experiment in which we use four different concentrations of a single catalyst. Now we can relate the four groups quantitatively by concentration of catalyst. We could still use the analysis of variance to see whether a change in concentration had any effect.

We might extend our first example using four different catalysts to study the effect of temperature as well. We could pick three different temperatures and determine the setting rate for each of the four catalysts. This would require a *two-way analysis of variance* to determine significant differences among the 12 setting times that we would obtain.

2) Dispersion analysis

Problem 1.25

On a pilot-plant for producing composite rocket propellant four batches of the same composition were made under the same process conditions with the two different burning-rate catalysts. From the obtained propellant experimental rocket motors were static fired. The following burning rates at 70 [bar] pressure were determined from the calculated burning rate laws:

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2) Dispersion analysis

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Finally, we might add a third independent variable, catalyst concentration, to type of catalyst and temperature of setting. We then would use a *three-way analysis of variance* to determine whether differences in means exist. Of course, as additional independent variables are added, the calculations become much more complex, so that they are better carried out on a digital computer.

Notation and arithmetic

Although the principles of analysis of variance are simple, the notation and arithmetic can be quite confusing at first contact. For this reason, we begin by discussing a few conventions in notation and arithmetic that we will use later. Suppose we have the following matrix:

Table 1.14 Data matrix

Column		1	2	.	.	.	j	.	.	.	J
	1	X_{11}	X_{12}	·	·	·	X_{1j}	·	·	·	X_{1J}
	2	X_{21}	X_{22}	·	·	·	X_{2j}	·	·	·	X_{2J}
R	.	·	·				·				·
	.	·	·				·				·
O	.	·	·				·				·
	i	X_{i1}	X_{i2}	·	·	·	X_{ij}	·	·	·	X_{iJ}
W	.	·	·				·				·
	.	·	·				·				·
	.	·	·				·				·
	I	X_{I1}	X_{I2}	·	·	·	X_{Ij}	·	·	·	X_{IJ}
Sums	I	$X_{\bullet 1}$	$X_{\bullet 2}$	·	·	·	$X_{\bullet j}$	·	·	·	$X_{\bullet J}$
Means		$\bar{X}_{\bullet 1}$	$\bar{X}_{\bullet 2}$	·	·	·	$\bar{X}_{\bullet j}$	·	·	·	$\bar{X}_{\bullet J}$

Each data point is subscripted, first to identify its column location and second to identify its row location. Thus, X_{32} (read "X" three two) is the data point in the third column and second row. Each column may be regarded as size I random sample drawn from the normal population. This matrix might represent the example of one-way analysis of variance given earlier. The columns would be the four catalysts and the rows would simply identify the succesive runs made at identical conditions.

In the two-way example, the columns would again be the four catalysts, the rows would be the three temperatures, and each X would be a single value of the setting time for a given temperature and catalyst. Thus, X_{32} is the setting time using the third catalyst and the second temperature.

We designate a general location in the matrix of data as X_{ij}, where i refers to the column, and j to the row. The sum of values in the i-th column is:

$$X_{i\bullet} = \sum_{j=1}^{J} X_{ij} \tag{1.106}$$

The dot refers to the direction that has been summed. The mean of the values in the i-th column is then:

$$\bar{X}_{i\bullet} = \frac{X_{i\bullet}}{J} \tag{1.107}$$

Similarly, the sum for any row j is:

$$X_{\bullet j} = \sum_{i=1}^{I} X_{ij} \tag{1.108}$$

ant the mean is:

$$\bar{X}_{\bullet j} = \frac{X_{\bullet j}}{I} \tag{1.109}$$

The sum of all the values in the matrix is designated by $\bar{X}_{\bullet\bullet}$ where:

$$X_{\bullet\bullet} = \sum_{i=1}^{I}\sum_{j=1}^{J} X_{ij} = \sum_{i=1}^{I} X_{i\bullet} = \sum_{j=1}^{J} X_{\bullet j} \tag{1.110}$$

The mean of all the values in the matrix is called the *grand mean* $\bar{X}_{\bullet\bullet}$ where:

$$\bar{X}_{\bullet\bullet} = \frac{X_{\bullet\bullet}}{IJ} \tag{1.111}$$

From here on, to simplify the equations, we will designate:

$$\sum_{i=1}^{I} = \sum_{i}; \quad \sum_{j=1}^{J} = \sum_{j}$$

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The same as before, capital letters denote random variable and the small ones the concrete value of the variable.

One-way analysis of variance

In one-way analysis of variance, we have several groups for which we wish to test equality of means. To apply the standard methods, we must assume that each group is normally distributed and that the population variance σ_X^2 is constant among the groups. In other words, one-way analysis of variance is used in situations when we want to test the J population means. The procedure is based on the assumption that each J group of observations is a random sample from normal population with its variance $\sigma_X^2 = \sigma^2$ that is mutual for all the groups.

Actually, analysis of variance may be applied where these criteria are not exactly met; but the procedure is based on these assumptions.

Suppose we wish to estimate the population variance. There are two possible estimates. First, we might use a pooled estimate such as that in an earlier section, in which we used the t-test on two means. A second method of estimating the population variance is to calculate the variance of the group means around the grand mean.

Pooled variance estimates

In the pooled estimate, we calculate the sample variance for each group (each column of data in a one-way analysis). Then we weigh each of these estimates by its degrees of freedom to obtain a pooled sample variance. For any column i, the sample variance is:

$$S_j^2 = \frac{\sum_i \left(X_{ij} - \bar{X}_{\bullet j}\right)^2}{I-1} \tag{1.112}$$

We have already asserted that $E\left(S_j^2\right) = \sigma^2$ for each j. We will assume for simplicity that each column contains the same number of values (i.e. J is constant). We have J estimates of the form of Eq. (1.112). To pool them, we weigh each by its degrees of freedom (I-1), add and divide by the total degrees of freedom J(I-1): The pooled sample variance[3)] is then:

$$S_p^2 = \frac{\sum_j \left[(I-1)S_j^2\right]}{J(I-1)} \tag{1.113}$$

Combining Eqs. (1.112) and (1.113) gives:

$$S_p^2 = \frac{\sum_j \left[\sum_i \left(X_{ij} - \bar{X}_{\bullet j}\right)^2\right]}{J(I-1)} = \frac{SS_W}{J(I-1)} = MS_W \tag{1.114}$$

In this equation, the term SS_W is refereed to as the the sum of squares within groups or error sum of squares. The quantity SS_W when divided by the appropriate degrees of freedom J(I-1) is referred to as the mean square or error mean square and is denoted by MS_W. As Eq. (1.114) is not particularly convenient for calculation purposes, it can be presented in the more usable form:

$$S_p^2 = \frac{\sum_i \sum_j \left(X_{ij}\right)^2 - \left(\sum_j X_{\bullet j}^2\right) \Big/ I}{J(I-1)} = \frac{SS_W}{J(I-1)} = MS_W \tag{1.115}$$

The pooled estimator of the population variance, S_p^2 , is an unbiased estimator for σ^2 regardless of whether the population means $\mu_1, \mu_2, \ldots, \mu_J$ are equal or not, because it takes into account deviations from each group mean $\bar{X}_{\bullet j}$, j=1, 2,..., J. Unbiasedness follows from Eq. (1.114) since:

$$E\left(S_p^2\right) = \frac{\sum_j E\left(\sum_i \left(X_{ij} - \bar{X}_{\bullet j}\right)^2 \Big/ (I-1)\right)}{J} = \frac{\sum_j E\left(S_j^2\right)}{J} = \sigma^2 \tag{1.116}$$

3) Pooled sample variance

Variance of group means

A second method of estimating the population variance σ^2 is to calculate the sample variance of the group means around the grand mean by use of:

$$S_{\bar{X}}^2 = \frac{\sum_j \left(\bar{X}_{\bullet j} - \bar{X}_{\bullet\bullet}\right)^2}{J-1} \tag{1.117}$$

If the group population means are all equal, then $S_{\bar{X}}^2$ is an unbiased estimate of the variance of the population mean $\sigma_{\bar{X}}^2$. To obtain an estimator of the population variance σ^2 recall that:

$$S_{\bar{X}}^2 = \frac{S_X^2}{I} \tag{1.118}$$

Combining Equs. (1.117) and (1.118) we have our second estimator of the population variance σ^2:

$$S^2 = \frac{I\sum_j \left(\bar{X}_{\bullet j} - \bar{X}_{\bullet\bullet}\right)^2}{J-1} = \frac{SS_B}{J-1} = MS_B \tag{1.119}$$

The quantities SS_B and MS_B are usually referred to as the between groups sum of squares and mean square for between groups, respectively. Eq. (1.119) is not suitable for practical calculations so it is transformed into the following expression:

$$S^2 = \frac{\left(\sum_j X_{\bullet j}^2\right) \Big/ I - X_{\bullet\bullet}^2 \Big/ IJ}{J-1} = \frac{SS_B}{J-1} = MS_B \tag{1.120}$$

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The estimator S^2 given in Eqs. (1.119) and (1.120) is an unbiased estimator of σ^2 only when the group population means are equal. If the population means $\mu_1, \mu_2, \ldots, \mu_J$ are not all equal then the estimator S^2 overestimates σ^2, that is $E(S^2) > \sigma^2$. The estimators S^2 and S_p^2 are linked by a very important identity given by:

$$\sum_j \sum_i \left(X_{ij} - \bar{X}_{\bullet\bullet}\right)^2 = I\sum_j \left(\bar{X}_{\bullet j} - \bar{X}_{\bullet\bullet}^2\right)^2 + \sum_j \sum_i \left(X_{ij} - \bar{X}_{\bullet j}\right)^2 \tag{1.121}$$

The left-hand side of Eq. (1.121) is usually referred to as the total sum of squares corrected for the mean and is denoted by SS_{TC}. Combining Eqs. (1.114), (1.119) and (1.121) gives:

$$SS_{TC} = SS_B + SS_W \tag{1.122}$$

In other words the total variations are partitioned into two components, a component SS_B that reflects variation among groups and a component SS_W that reflects experimental error or sampling variation. The degrees of freedom associated with SS_{TC} are also partitioned into the degrees of freedom associated with SS_B and SS_W, i.e., IJ-1=(J-1)+J(I-1). If the means $\mu_1, \mu_2, \ldots, \mu_J$ are all equal then S_p^2 and S^2 are independent so that the random variable:

$$F = \frac{S^2}{S_p^2} = \frac{\frac{SS_B}{J-1}}{\frac{SS_W}{J(I-1)}} = \frac{MS_B}{MS_W} \tag{1.123}$$

has an F-distribution with J-1 and J(I-1) degrees of freedom. The Eq. (1.123) illustrates the variance analysis basis; two variances were compared in order to test the mean equality. Thus under H_0: $\mu_1=\mu_2=...=\mu_J$ we would expect the value of F to be close to 1. If H_0 is not true then the value S^2 would tend to be larger than S_p^2, which would force F to be larger than 1. Consequently, based on the data, the hypothesis H_0 would be rejected if the computed F-value, is too large. That is, the rejection region is of the form:

$$F \succ F_{k_1,k_2.1-\alpha}$$

where:

k_1=J-1 and k_2=J(I-1)

Fisher introduced the following table for a clear presentation of variance analysis results:

Table 1.15 One-way analysis of variance

Source of variation	f	Sum of squares	Mean square	Test statistic
Between columns	J-1	$SS_B = \frac{\sum_j X_{\bullet j}^2}{I} - \frac{X_{\bullet\bullet}^2}{IJ}$	$MS_B = \frac{SS_B}{J-1}$	$\frac{MS_B}{MS_W}$
Within columns – error	J(I-1)	$SS_W = \sum_i \sum_j X_{ij}^2 - \frac{X_{\bullet j}^2}{I}$	$MS_W = \frac{SS_W}{J(I-1)}$	–
Total	JI-1	$SS_T = \sum_i \sum_j X_{ij}^2 - \frac{X_{\bullet\bullet}^2}{IJ}$	–	–

Example 1.23 [11]

A quantity of each of three chemical fertilizers was applied to three groups of five corn plants each, with all plants growing under identical conditions of temperature, rainfall, soil, seed, etc. From the following measures of corn growth (height after one month), determine whether there is any reason for one fertilizer to be better than another:

	Fertilizer		
	No1	No2	No3
	23	16	18
	21	23	22
Height	24	20	25
	17	21	21
	19	18	20
	$X_{\bullet 1}$=104	$X_{\bullet 2}$=98	$X_{\bullet 3}$=106
	$\bar{X}_{\bullet 1}$=20.8	$\bar{X}_{\bullet 2}$=19.6	$\bar{X}_{\bullet 3}$=21.2

First, we evaluate the summations:

$$\sum_i \sum_j \left(X_{ij}\right)^2 = 23^2 + 21^2 + 24^2 + 17^2 + 19^2 + 16^2 + \ldots + 20^2 = 6420$$

$$\sum_j X_{\bullet j}^2 = 104^2 + 98^2 + 106^2 = 31656$$

$$X_{\bullet\bullet}^2 = (104 + 98 + 106)^2 = 94864$$

Then from Equs. (1.115) and (1.120), we get:

$$S_p^2 = \frac{6420 - \frac{31656}{5}}{3(5-1)} = 7.40;\ k_1 = 12$$

$$S^2 = \frac{\frac{31656}{5} - \frac{94864}{3\times 5}}{3-1} = 3.50;\ k_2 = 2$$

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From this point, we may use the 7-step test procedure:

1. Assume underlying normally distributed populations for each fertilizer group. All groups have constant population variance $\sigma_1^2 = \sigma_2^2 = \sigma_3^2$.
2. H_0:μ_1=μ_2=μ_3 H_1: H_0 is not true
3. Test statistic: $F = \frac{S^2}{S_p^2}$
4. Let α=0.05
5. We reject H_0 if F is higher than $F_{0.95(2;12)}$=3.88
6. F=3.50/7.40=0.47
7. Accept H_0 since 0.47<3.88

Thus, there is no reason to believe that one fertilizer promotes growth more than another. Generally speaking, analysis-of-variance problems are not solved in the form used in this example. A standard form called the analysis-of-variance table has been developed, and it is particularly useful for more complex problems:

$$SS_T = \sum_i \sum_j X_{ij}^2 - \frac{X_{\bullet\bullet}^2}{IJ} = 6420 - \frac{94864}{3\times 5} = 95.8$$

$$SS_B = \frac{\sum_j X_{\bullet j}^2}{I} - \frac{X_{\bullet\bullet}^2}{IJ} = \frac{31656}{5} - \frac{94864}{3\times 5} = 7.0$$

$$SS_W = SS_T - SS_B = 95.8 - 7.0 = 88.8$$

Table 1.16 Analysis of variance

Source of variation	f	SS	MS	F	$F_{2;12;0.95}$
Between groups	2	7.0	3.50	0.47	3.88
Within groups	12	88.8	7.40	–	–
Total	14	95.8	–	–	–

Example 1.24 [4]

The amount of fluoride in the local water supply was determined by the four colorimetric methods in a comparative study *A, B, C* and *D*. Five replications were made for each test. To preclude bias from variations in the sample over the time required for the analysis, all samples were taken from a single 10-gal carboxy of water. The results in ppm are:

A: 2; 3; 6; 5; 4;
B: 5; 4; 4; 2; 3;
C: 1; 3; 2; 4; 4;
D: 2; 1; 1; 2; 1.

a) Are the methods equivalent? Use the 5% significance level.
b) What are the 95% confidence limits on the values obtained from each method.

a) We first calculate the required sums and squares:

$$X_{\bullet A} = 20; \quad \sum_i X_{iA}^2 = 90; \quad \bar{X}_{\bullet A} = 4.0; \quad \sum_j X_{\bullet j}^2 = 969;$$

$$X_{\bullet B} = 18; \quad \sum_i X_{iB}^2 = 70; \quad \bar{X}_{\bullet B} = 3.6; \quad \sum_i \sum_j X_{ij}^2 = 217;$$

$$X_{\bullet C} = 14; \quad \sum_i X_{iC}^2 = 46; \quad \bar{X}_{\bullet C} = 2.8; \quad X_{\bullet\bullet} = 59;$$

$$X_D = 7; \quad \sum_i X_{iD}^2 = 11; \quad \bar{X}_D = 1.4.$$

We calculate the pooled sample variance:

$$S_p^2 = \frac{\sum_i \sum_j X_{ij}^2 - \frac{\sum_j X_{\bullet j}^2}{I}}{J(I-1)} = \frac{217-193.8}{16} = 1.45$$

$$S^2 = \frac{\frac{\sum_j X_{\bullet j}^2}{I} - \frac{X_{\bullet\bullet}^2}{IJ}}{J-1} = \frac{\frac{969}{5} - \frac{59^2}{20}}{3} = 6.58$$

$$F = \frac{S^2}{S_p^2} = \frac{6.58}{1.45} = 4.54 \succ F_{3;16;0.95} = 3.24$$

Table 1.17 Analysis of variance

Source of variation	f	SS	MS	F	$F_{3;16.0,95}$
Between groups	3	19.75	6.58	4.54	3.24
Error	16	23.20	1.45	–	–
Total	19	42.95	–	–	–

Based on F test reject H_0 and conclude that there are significant differences among the applied methods.

b) For 16 degrees of freedom, at the 95% confidence level, $t_{16;0.975}$=2.120 and $t_{16;0.025}$=-2.120. The standard error is found as before:

$$S_{\bar{p}} = \frac{S_p}{\sqrt{I}} = \frac{\sqrt{1.45}}{\sqrt{5}} = 0.5386$$

The confidence limits for the means corresponding to the four colorimetric methods are:
μA : 4.0±2.12 × 0.5386 ⇒2.96<μA<5.04
μB : 3.6±2.12 × 0.5386 ⇒2.56<μB<4.64
μC : 2.8±2.12 × 0.5386 ⇒1.76<μC<3.84
μD : 1.4±2.12 × 0.5386 ⇒0.36<μD<2.44

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Example 1.25 [12]
The conductivity of four different coatings on cathode tubes was tested. As only four types of coatings were tested, we had a one-way experiment on four levels. The mentioned levels were qualitative as we had no quantitative measure for coating types. Five cathode tubes were tested for each coating. The sequence of conductivity measurements was completely random. The obtained results are given in the following table:

Coating			
I	II	III	IV
56	64	45	42
55	61	46	39
62	50	45	45
59	55	39	43
60	56	43	41

If we subtract 50 from each value we shall obtain coded values, which to a great extent will simplify the arithmetic and has no influence on the F-statistic.

I	II	III	IV	
6	14	–5	–8	
5	11	–4	–11	
12	0	–5	–5	
9	5	–11	–7	
10	6	–7	–9	
$X_{\bullet j}$ = 42;	36;	–32;	–40;	$X_{\bullet\bullet}$ = 6
I 5;	5;	5;	5;	I × J=20
$\sum X_{ij}^2$ = 386	378	236	340	$\sum\sum X_{ij}^2$ = 1340

$$SS_T = \sum_i \sum_j X_{ij}^2 - \frac{X_{\bullet\bullet}^2}{IJ} = 1340 - \frac{6^2}{20} = 1338.2$$

$$SS_B = \frac{\sum_j X_{\bullet j}^2}{I} - \frac{X_{\bullet\bullet}^2}{IJ} = \frac{5684}{5} - \frac{6^2}{20} = 1135.0$$

$$SS_W = SS_T - SS_B = 1338.2 - 1135.0 = 203.2$$

Table 1.18 Analysis of variance

Source of variation	f	SS	MS	F	$F_{3;16;0.95}$
Between groups	3	1135.0	378.3	29.8	3.24
Error	16	203.2	12.7	–	–
Total	19	1338.2	–	–	–

Since the calculated F-criterion value is greater than the tabulated one, the null hypothesis can be rejected with 95% confidence level, i.e. the alternative hypothesis that there is a statistical difference between the used coatings is accepted.

Model for one-way analysis of variance

Up to now the technique of calculations in analysis of variance has been analyzed in more detail. Now let us briefly consider the analysis of variance theory. Let us consider the model for a one-way analysis of variance. Here it is assumed that the columns of data are J-random samples from J-independent normal populations with means $\mu_1,\mu_2,...,\mu_J$, and common variance σ^2. The one-way analysis of variance technique will give us a procedure for testing the hypothesis: H_0: $\mu_1=\mu_2=...=\mu_J$;against the alternative H_1: at least two μ_i not equal. The statistical model gives us the structure of each observation in the I×J matrix:

$$X_{ij}=\mu_j+\varepsilon_{ij} \tag{1.124}$$

This model says that the dependent variable X_{ij} is made up of two parts: the first part μ that is the mean of the population corresponding to the j-th column (population) and the second part, ε_{ij}, the random experimental error that is taken to have

mean 0, i.e., $E(\varepsilon_{ij})=0$. This must be the case since $E(X_{ij})=\mu_{ij}$. The model in Eq. (1.124) can be written as:

$$X_{ij}=\mu_j+\mu-\mu+\varepsilon_{ij}=\mu+(\mu_j-\mu)+\varepsilon_{ij}=\mu+\alpha_j+\varepsilon_{ij} \tag{1.125}$$

where:

$\mu = \bar{\mu} = \dfrac{\sum_j \mu_j}{J}$ is called the grand population mean

$\alpha_j = \mu_j - \bar{\mu}$ is called the effect of the j-th population.

Eq. (1.125) states that any experimental value is the sum of a term representing the general location of the grand population mean plus a term α_j showing the displacement of a given population from the general location, plus a term giving the random experimental error ε_{ij} of the particular observation.

The ε_{ij} are independent and normally distributed with mean 0 and variance σ^2, and it is the result of random fluctuations in the process and measurement errors. The population grand mean may be considered the main addend for X_{ij}.

The α_j is the column contribution (the contribution that arises if the column population means are different so that each column mean would be different from the grand population).

This means that α_j has already been defined as:

$$\alpha_j = \mu_j - \bar{\mu} \tag{1.126}$$

Note that α_j is a constant for any column in a specific analysis of variance as shown in Fig. 1.17.

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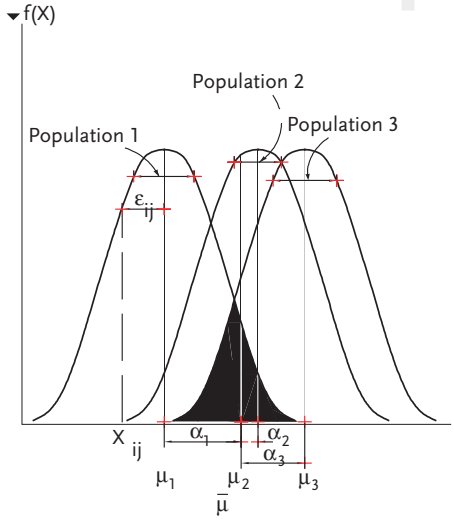

Figure 1.17 Model defines terms for one-way AHOVA

If all the columns are equal, $\mu=\mu_j$ and $\alpha_j=0$. Therefore, the hypothesis $H_0:\mu_1=\mu_2=...=\mu_J$ is the same as the hypothesis H_1: $\alpha_j=0$ for all j. One is generally interested in the hypothesis H_0: $\alpha_1=\alpha_2=...=\alpha_J=0$, which states that there is no population or column effect. This means that the variation in $\bar{X}_{\bullet 1}, \bar{X}_{\bullet 2}, ..., \bar{X}_{\bullet j}$ is due to experimental error and not to any difference in population means. To test the hypothesis H_0 we use the F-statistic given in Eq. (1.123):

$$F = \frac{\frac{SS_B}{J-1}}{\frac{SS_W}{J(I-1)}} = \frac{MS_B}{MS_W}$$

The rejection region at significance level α is $F>F_{J-1,J(I-1),1-\alpha}$.

Recall from Eq. (1.116) that:

$$E\left(S_p^2\right) = E(MS_W) = \sigma^2$$

Furthermore, if H_0: $\alpha_1=\alpha_2=...=\alpha_J$ is true, then:

$E(S^2)=E(MS_B)=\sigma^2$.

However, if H_0 is not true then $E(S^2)\neq\sigma^2$. To show this, consider:

$$E\left(S^2\right) = \sigma^2 + \frac{I}{J-1}\sum_j \alpha_j^2 \tag{1.127}$$

Eq. (1.116) indicates that the pooled estimate S_p^2 is unbiased, and Eq. (1.127) biased estimation S^2. For the F-test we use the unbiased estimate of σ^2 in the denominator of the F-ratio and the biased estimate in the numerator.

Two-way analysis of variance

If we desire to study the effects of two independent variables (factors) on one dependent factor, we will have to use a two-way analysis of variance. For this case the columns represent various values or levels of one independent factor and the rows represent levels or values of the other independent factor. Each entry in the matrix of data points then represents one of the possible combinations of the two independent factors and how it affects the dependent factor. Here, we will consider the case of only one observation per data point. We now have two hypotheses to test. First, we wish to determine whether variation in the column variable affects the column means. Secondly, we want to know whether variation in the row variable has an effect on the row means. To test the first hypothesis, we calculate a "between columns" sum of squares: and to test the second hypothesis, we calculate a "between rows" sum of squares. The between-rows mean square is an estimate of the population variance, providing that the row means are equal. If they are not equal, then the expected value of the between-rows mean square is higher than the population variance. Therefore, if we compare the between-rows mean square with another unbiased estimate of the population variance, we can construct an F test to determine whether the row variable has an effect. Definitional and calculational formulas for these quantities are given in Table 1.19.

Table 1.19 Two-way analysis of variance

Source of variation	Degrees of freedom	Sum of squares-definition	Sum of squares-practical calculation	Mean squares	Test statistic
Between columns	J-1	$SS_C = I\sum_j \left(\bar{X}_{\bullet j} - \bar{X}_{\bullet\bullet}\right)^2$	$SS_C = \frac{\sum_j X_{\bullet j}^2}{I} - \frac{X_{\bullet\bullet}^2}{IJ}$	$MS_C = \frac{SS_C}{J-1}$	$\frac{MS_C}{MS_E}$
Between rows	I-1	$SS_R = J\sum_i \left(\bar{X}_{i\bullet} - \bar{X}_{\bullet\bullet}\right)^2$	$SS_R = \frac{\sum_i X_{i\bullet}^2}{J} - \frac{X_{\bullet\bullet}^2}{IJ}$	$MS_R = \frac{SS_R}{I-1}$	$\frac{MS_R}{MS_E}$
Residual variance-error	(I-1)(J-1)	$SS_E = \sum_i \sum_j \left(X_{ij} - \bar{X}_{i\bullet} - \bar{X}_{\bullet j} + \bar{X}_{\bullet\bullet}\right)^2$	$SS_E = SS_T - SS_C - SS_R$	$MS_E = \frac{SS_E}{(I-1)(J-1)}$	–
Total	IJ-1	$SS_T = \sum_i \sum_j \left(X_{ij} - \bar{X}_{\bullet\bullet}\right)^2$	$SS_T = \sum_i \sum_j X_{ij}^2 - \frac{X_{\bullet\bullet}^2}{IJ}$	–	–

We note from Table 1.19 that the sums of squares between rows and between columns do not add up to the defined total sum of squares. The difference is called the sum of squares for error, since it arises from the experimental error present in each observation. Statistical theory shows that this error term is an unbiased estimate of the population variance, regardless of whether the hypotheses are true or not. Therefore, we construct an F-ratio using the between-rows mean square divided by the mean square for error. Similarly, to test the column effects, the F-ratio is the between-columns mean square divided by the mean square for error. We will reject the hypothesis of no difference in means when these F-ratios become too much greater than 1. The ratios would be 1 if all the means were identical; and the assumptions of normality and random sampling hold. Now let us try the following example that illustrates two-way analysis of variance.

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Example 1.26 [11]

Determine whether the type of catalyst or temperature has any effect on the setting time of a new plastic from the following data. The measured variable-response is elapsed setting time (in minutes) to a uniform criterion of hardness.

Temperature	Catalyst				
[°C]	No1	No2	No3	No4	
25	25	28	22	24	$X_{1\bullet} = 99$
50	27	29	23	23	$X_{2\bullet} = 102$
75	30	32	26	29	$X_{3\bullet} = 117$
	$X_{\bullet 1} = 82$	$X_{\bullet 2} = 89$	$X_{\bullet 3} = 71$	$X_{\bullet 4} = 76$	$X_{\bullet\bullet} = 318$

The analysis-of-variance table is constructed from the following quantities calculated from the data:

I=3; J=4;

$$\sum_j X_{\bullet j}^2 = 82^2 + 89^2 + 71^2 + 76^2 = 25462$$

$$X_{\bullet\bullet}^2 = 318^2 = 101124$$

$$\sum_i X_{i\bullet}^2 = 99^2 + 102^2 + 117^2 = 33894$$

$$\sum_i \sum_j X_{ij}^2 = 25^2 + 27^2 + \ldots + 23^2 + 29^2 = 8538$$

Inspection of Table 1.20 shows that we reject the hypotheses of no effect of the column variable or the row variable. Both type of catalyst and temperature seem to have an effect. Of course, we have made only a preliminary survey. We would now take more data to determine which catalyst was best and to evaluate a quantitative relationship on the temperature effect.

Table 1.20 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Between columns	3	60.3	20.1	28.7	$F_{3;6;0.95}$=4.76
Between rows	2	46.5	23.3	33.3	$F_{2;6;0.95}$=5.14
Error	6	4.2	0.7	–	–
Total	11	111.0	–	–	–

Example 1.27 [4]

In an experiment to determine the effects of varying the reflux ratio on the number of required stages X_{ij} used in the separation of benzene and toluene, four different lab groups used the same four reflux ratios with the following results:

	Reflux ratio				
Lab group	No1	No2	No3	No4	
1	11.4	9.2	7.5	6.2	$X_{1\bullet} = 34.3$
2	10.7	8.6	8.3	5.9	$X_{2\bullet} = 33.5$
3	11.9	8.7	9.3	5.4	$X_{3\bullet} = 35.3$
4	9.9	9.0	7.1	5.6	$X_{3\bullet} = 31.6$
	$X_{\bullet 1} = 43.9$	$X_{\bullet 2} = 35.5$	$X_{\bullet 3} = 32.2$	$X_{\bullet 4} = 23.1$	$X_{\bullet\bullet} = 134.7$

$$SS_T = \sum_i \sum_j X_{ij}^2 - \frac{X_{\bullet\bullet}^2}{IJ} = 1195.7 - \frac{18144.09}{4 \times 4} = 61.1644$$

$$SS_C = \frac{\sum_j X_{\bullet j}^2}{I} - \frac{X_{\bullet\bullet}^2}{IJ} = \frac{4757.91}{4} - 1134.0056 = 55.4719$$

$$SS_R = \frac{\sum_i X_{i\bullet}^2}{J} - \frac{X_{\bullet\bullet}^2}{IJ} = \frac{4543.39}{4} - 1134.0056 = 1.8419$$

$SS_E = SS_T - SS_C - SS_R = 3.8506$

Table 1.21 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Reflux ratio	3	55.4719	18.4906	43.222	$F_{3;9;0.95}=3.86$
Lab group	3	1.8419	0.6139	1.435	$F_{3;9;0.95}=3.86$
Error	9	3.8506	0.4278	–	–
Total	15	61.1644	–	–	–

When the calculated values of F are compared to $F_{3;9;0.95}=3.86$ it is seen at once that the differences in the number of required stages is significantly affected by the reflux ratio. No significant differences between lab groups were found.

Example 1.28 [12]

Determine whether car tires of different producers have different wear-out rates after 40.000 km. Mark the car tire types as: A, B, C and D. The wear-out of car tyres will be tested on four types of cars. As each car needs four tyres, the experiment will test 16 tyres, four of each type. The experiment was done by each car having one type of each tyre. The sequence of putting tyres on wheels was completely random in order to center and eliminate the effect of differences between the wheels, if it exists at all.

Apart from determining whether the car-tyre type has a significant influence on wear-out, decide how much the car type influences it. The obtained data have been given in millimeters.

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Table 1.22 Analysis of variance

Car type	Tire type				$X_{\bullet j}$
	A	B	C	D	
I	17	14	12	13	56
II	14	14	12	11	51
III	13	13	10	11	47
IV	13	8	9	9	39
$X_{\bullet j}$	57	49	43	44	$193 = X_{\bullet\bullet}$
$\sum X_{ij}^2$	823	625	469	492	$\sum_i \sum_j X_{ij}^2 = 2409$

$$SS_T = \sum_i \sum_j X_{ij}^2 - \frac{X_{\bullet\bullet}^2}{IJ} = 2409 - \frac{37249}{16} = 80.94$$

$$SS_C = \frac{\sum_j X_{\bullet j}^2}{I} - \frac{X_{\bullet\bullet}^2}{IJ} = \frac{9435}{4} - \frac{37249}{16} = 30.69$$

$$SS_R = \frac{\sum_i X_{i\bullet}^2}{J} - \frac{X_{\bullet\bullet}^2}{IJ} = \frac{9467}{4} - \frac{37249}{16} = 38.69$$

$SS_E = SS_T - SS_C - SS_R = 11.56$

Table 1.23 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Tire type	3	30.69	10.23	7.99	$F_{3;9;0.95}=3.86$
Car type	3	38.69	12.90	10.08	$F_{3;9;0.95}=3.86$
Error	9	11.56	1.28	–	–
Total	15	80.94	–	–	–

The above results allow us to conclude with 95% confidence that different car tyre types and different types of cars have important influences on tyre wear-out. The same conclusion can be drawn even at 99% confidence level as $F_{3;9;0.99}=6.99$.

Model for two-way analysis of variance

For a two-way analysis of variance the assumed model is:

$$X_{ij} = \mu + \alpha_i + \beta_j + \varepsilon_{ij} \tag{1.128}$$

where:

X_{ij} is assumed to come from a normal population with mean μ_{ij} and variance σ^2;

$$\mu = \bar{\mu}_{\bullet\bullet} = \frac{\sum_i \sum_j \mu_{ij}}{IJ};$$

$$\alpha_i = \bar{\mu}_{i\bullet} - \bar{\mu}_{\bullet\bullet};$$
$$\beta_j = \bar{\mu}_{\bullet j} - \bar{\mu}_{\bullet\bullet};$$

$$\bar{\mu}_{i\bullet} = \frac{\sum_j \mu_{ij}}{J} \quad ; \qquad \bar{\mu}_{\bullet j} = \frac{\sum_i \mu_{ij}}{I}.$$

The parameter μ is the contribution of the grand mean, α_i is the contribution of the i-th level of the row variable, β_j is the contribution of the j-th level of the column variable, and ε_{ij} is the random experimental error. The model in Eq. (1.128) does not contain what is usually referred to as row-column interaction; that is, the row and

column effects are additive. The restrictions (or assumptions) related to the model in Eq. (1.128) are:

$$\sum_i \alpha_i = 0 \qquad \text{and} \qquad \sum_j \beta_j = 0$$

These follow from:

$$\alpha_i = \bar{\mu}_{i\bullet} - \bar{\mu}_{\bullet\bullet} \qquad \textit{and} \qquad \beta_j = \bar{\mu}_{\bullet j} - \bar{\mu}_{\bullet\bullet} .$$

Two hypotheses related to the model are:

H_0: $\mu_{1(}=\mu_{2(}=...=\mu_I$ and
H_0: $\mu_{(1}=\mu_{(2}=...=\mu_J$

The first hypothesis says that there is no row effect, that is, the means across rows have the same value. Similarly, the second hypothesis says there is no column effect. The two hypotheses above can be written equivalently as:

$$H_0: \alpha_1=\alpha_2=...=\alpha_I=0 \tag{1.129}$$

$$H_0: \beta_1=\beta_2=...=\beta_J=0 \tag{1.130}$$

Furthermore, it can be shown (just as for the one-way model) that the expected mean squares are:

$$E(MS_R) = \sigma^2 + \frac{J}{I-1}\sum_i \alpha_i^2$$

$$E(MS_C) = \sigma^2 + \frac{I}{J-1}\sum_j \beta_j^2$$

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$E(MS_E)=\sigma^2$

Thus, to test H_0: all α_i=0, we use the F-statistic with I-1 and (I-1)(J-1) degrees of freedom.

$$F = \frac{MS_R}{MS_E}$$

The rejection region is: $F>F_{(I-1),(I-1)(J-1),1-\alpha}$. Similarly, to test H_0 all β_j=0, we use F-statistic with J-1 and (I-1)(J-1) degrees of freedom.

$$F = \frac{MS_C}{MS_E}$$

The rejection region is: $F>F_{(J-1),(I-1)(J-1),1-\alpha}$.

Confidence intervals and tests of hypotheses

In the two-way model for analysis of variance:

$X_{ij}=\mu+\alpha_i+\beta_j+\varepsilon_{ij}$

it may be of interest to compare the effects due to two rows, say α_1 and α_2, if: H_0: all α_i=0 has been rejected. The mean for the i-th row is:

$$X_{i\bullet} = \mu + \alpha_i + \bar{\beta}_\bullet + \bar{\varepsilon}_{i\bullet}$$

where:

$$\bar{\beta}_\bullet = \frac{\sum_j \beta_j}{J} = 0 \qquad \text{and} \qquad \bar{\varepsilon}_{i\bullet} = \frac{\sum_j \varepsilon_{ij}}{J},$$

since:

$$\bar{X}_{1\bullet} - \bar{X}_{2\bullet} = \alpha_1 - \alpha_2 + \bar{\varepsilon}_{1\bullet} - \bar{\varepsilon}_{2\bullet} \qquad \textit{and} \qquad E\left(\bar{X}_{1\bullet} - \bar{X}_{2\bullet}\right) = \alpha_1 - \alpha_2$$

since:

$E\left(\varepsilon_{ij}\right) = 0$ for all i and j

Now MS_E is an unbiased estimator for σ^2 and the random variable:

$$T = \frac{\bar{X}_{1\bullet} - \bar{X}_{2\bullet} - (\alpha_1 - \alpha_2)}{S_{\bar{X}_1 - \bar{X}_2}} = \frac{\bar{X}_{1\bullet} - \bar{X}_{2\bullet} - (\alpha_1 - \alpha_2)}{\sqrt{\hat{\sigma}^2\left(\frac{1}{J} + \frac{1}{J}\right)}} = \frac{\bar{X}_{1\bullet} - \bar{X}_{2\bullet} - (\alpha_1 - \alpha_2)}{\sqrt{MS_E \frac{2}{J}}} \tag{1.131}$$

has a t-distribution with (I-1)(J-1) degrees of freedom.

Referring to Eq. (1.74) (1-α) 100% confidence interval for α_1-α_2 can be constructed:

$$\bar{X}_{1\bullet} - \bar{X}_{2\bullet} \pm t_{k,1-\alpha/2}\left(\frac{2MS_E}{J}\right)^{0.5} \tag{1.132}$$

where:

k=(I-1)(J-1).

If these confidence limits cover the value zero then we accept H_0: α_1=α_2, that is, there is no significant difference between the effects due to rows 1 and 2.

They can also be used for any pair of column effects if the difference of the sample means is replaced by $\bar{X}_{\bullet 1} - \bar{X}_{\bullet 2}$ and J is replaced with I.

Consider the random variable $\sum_i c_i \bar{X}_i$, where c_1, c_2,..., c_I are any constants. It is not difficult to show that $\sum_i c_i \bar{X}_i$ has mean $\sum_i c_i(\mu + \alpha_i)$ *and variance* $\sum_i c_i^2 \sigma^2 / J$. Now if we assume $\sum_i c_i = 0$, then the mean becomes:

$$\sum_i c_i \alpha_i$$

since:

$$\sum_i c_i \mu = \mu \sum_i c_i = 0$$

The linear combination $\sum_i c_i \alpha_i$ is called a *contrast* if $\sum_i c_i = 0$.

Let $L=\sum_i c_i\bar{X}_{i\bullet}$ be a contrast. Then L has a normal distribution with mean $\sum_i c_i\alpha_i$ and variance $\sum_i c_i^2\sigma^2/J$. Thus the random variable:

$$T=\frac{L-\mu_L}{\sigma_L}=\frac{\sum_i c_i\bar{X}_{i\bullet}-\sum_i c_i\alpha_i}{\sqrt{\frac{MS_E}{J}\sum c_i^2}} \tag{1.133}$$

has a t-distribution with (I-1)(J-1) degrees of freedom. From this we obtain a (1-α) × 100% confidence interval for the contrast $\sum_i c_i\alpha_i$:

$$\sum_i c_i\bar{X}_{i\bullet}-t_{k,1-\alpha/2}\left(\frac{MS_E}{J}\sum_i c_i^2\right)^{0.5}\prec\sum_i c_i\alpha_i\prec\sum_i c_i\bar{X}_{i\bullet}+t_{k,1-\alpha/2}\left(\frac{MS_E}{J}\sum_i c_i^2\right)^{0.5} \tag{1.134}$$

where:

k=(I-1)(J-1).

Similarly, for the contrast $\sum_j a_j\beta_j$:

$$\sum_j a_j\bar{X}_{\bullet j}-t_{k,1-\alpha/2}\left(\frac{MS_E}{I}\sum_j a_j^2\right)^{0.5}\prec\sum_j a_j\beta_j\prec\sum_j a_j\bar{X}_{\bullet j}+t_{k,1-\alpha/2}\left(\frac{MS_E}{I}\sum_j a_j^2\right)^{0.5} \tag{1.135}$$

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Example 1.29

Using the results of analysis of variance in Example 1.27 determine the 95% confidence interval for contrast β_1-β_2.

For the contrast we need to use the interval given in Eq. (1.135). Since:

α_1=1, α_2=-1; MS_E=0.4278; I=4; k=(I-1)(J-1)=9; $t_{9;0.975}$=2.262; $\bar{X}_{\bullet 1}$ =10.475; $\bar{X}_{\bullet 2}$ = 8.875,

the 95% confidence interval are:

$$\bar{X}_{1\bullet}-\bar{X}_{2\bullet}\pm 2.62\left(\frac{0.4278}{4}(1+1)\right)^{0.5}=10.475-8.875\pm 1.046$$

$$=1.600\pm 1.046\Rightarrow(0.544;2.646)$$

Since the confidence interval does not contain zero we can say that β_1-β_2>0 or β_1>β_2 or that the effect from the first reflux ratio is greater than that from the second reflux ratio. Confidence intervals can also be constructed for contrasts in the one-way model $X_{ij}=\mu+\alpha_j+\varepsilon_{ij}$. Confidence limits are:

$$\sum_j c_j \alpha_j = \sum_j c_j \left(\mu_j - \mu\right) = \sum_j c_j \mu_j$$

at the α level of significance is:

$$\sum_j c_j \bar{X}_{\bullet j} \pm t_{J(I-1);1-\alpha/2} \left(\frac{MS_E}{J(I-1)} \frac{\sum_j c_j^2}{I} \right)^{0.5} \tag{1.136}$$

Example 1.30

For the fluoride data of Example 1.24 determine a 95% confidence interval on α_A-α_B. The contrast is:

α_A-α_B=μ_A-μ-(μ_B-μ)=μ_A-μ_B.

From the results of Example 1.24 we have: $\bar{X}_{\bullet A} = 4.0$; $\bar{X}_{\bullet B} = 3.6$; J(I-1)=4(5-1)=16; MS_E=1.45; C_A=1; C_B=-1 and $t_{16;0.975}$=2.120. The 95% confidence limits on μ_A-μ_B are, from Eq. (1.136):

$$4.0 - 3.6 \pm 2.120 \left(\frac{1.45}{16} \left(\frac{1}{5} + \frac{1}{5} \right) \right)^{0.5} = 0.4 \pm 2.120 \times 0.1904 \Rightarrow (-0.0036; 0.8036)$$

Since the interval contains zero we conclude that there is no difference between methods A and B.

Interaction

Two-way analysis of variance (and higher classifications) leads to the presence of interactions. If, for example, an additive A is added to a lube oil stock to improve its resistance to oxidation and another additive, B, is added to inhibit corrosion by the stock under load or stress, it is entirely possible that the performance of the lube oil in a standard ball-and-socket wear test will be different from that expected if only one additive has present. In other words, the presence of one additive may adversely or helpfully affect the action of the other additive in modifying the properties of the lube oil. The same phenomenon is clearly evident in a composite rocket propellant where the catalyst effect on burning rate of the propellant drastically depends on the influence of fine oxidizer particles. These are termed antagonistic and synergistic effects, respectively. It is important to consider the presence of such interactions in any treatment of multiply classified data. To do this, the two-way analysis of variance table is set up as shown in Table 1.24.

In two-way analysis of variance with no replications, interaction of factors was part of the experimental error or more precisely of the residual variance. To separate the interaction from the residual variance or from experimental error variance, it is necessary to replicate *design point,* i.e. all combinations of rows and columns k times.

Table 1.24 Two-way analysis of variance with interactions

Source of variation	Degrees of freedom	Sum of squares-definition	Sum of squares-practical calculation	Mean squares	Test statistic
Between columns	J-1	$IK\sum_j \left(\bar{X}_{\bullet j\bullet} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_C = \frac{\sum_j X^2_{\bullet j\bullet}}{IK} - \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_C = \frac{SS_C}{J-1}$	$F = \frac{MS_C}{MS_E}$
Between rows	I-1	$JK\sum_i \left(\bar{X}_{i\bullet\bullet} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_R = \frac{\sum_i X^2_{i\bullet\bullet}}{JK} - \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_R = \frac{SS_R}{I-1}$	$F = \frac{MS_R}{MS_E}$
Interaction columns-rows	(J-1)(I-1)	$K\sum_i\sum_j \left(\bar{X}_{ij\bullet} - \bar{X}_{i\bullet\bullet} - \bar{X}_{\bullet j\bullet} + \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_{CR} = \frac{\sum_i\sum_j X^2_{ij\bullet}}{K} - \frac{\sum_i X^2_{i\bullet\bullet}}{JK} - \frac{\sum_j X^2_{\bullet j\bullet}}{IK} + \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_{CR} = \frac{SS_{CR}}{(I-1)(J-1)}$	$F = \frac{MS_{CR}}{MS_E}$
Error	IJ(K-1)	$\sum_i\sum_j\sum_k \left(X_{ijk} - \bar{X}_{i\bullet\bullet} - \bar{X}_{\bullet j\bullet} + \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_E = SS_T - SS_C - SS_R - SS_{CR}$	–	–
Total	IJK-1	$\sum_i\sum_j\sum_k \left(X_{ijk} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$\sum_i\sum_j\sum_k X^2_{ijk} - X^2_{\bullet\bullet\bullet}/IJK$	–	–

Three-way analysis of variance

Based on models and assumptions of one-way and two-way analyses of variance with or without replications of design points, it is possible to generalize for multiple-way analysis of variance. It is of interest to present the three-way analysis of variance for it is used quite often. In the case of a three-way analysis of variance the total number of observations is N=IxJxKxL, where I, J and K are numbers of levels or columns, rows and layers. L is the number of design-point replications or the number of observations in cells. Fig. 1.18 shows the tridimensional arrangement of columns, rows and layers.

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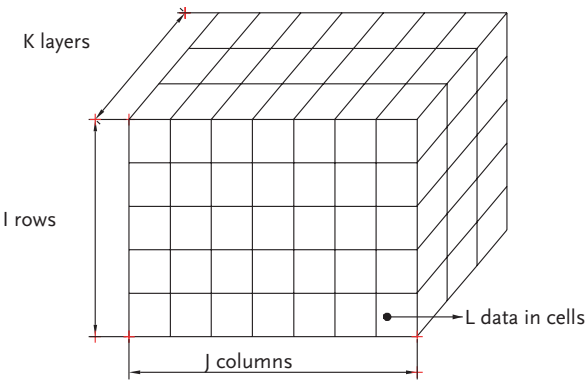

Figure 1.18 Arrangement of data in columns, rows and layers to represent three-way ANOVA

In calculation for the three-way analysis without replicating L=1, the same conventions in the notations were followed as for one and two-way analysis of variance.

$$X_{\bullet\bullet\bullet} = \sum_i \sum_j \sum_k X_{ijk}; \quad \bar{X}_{\bullet\bullet\bullet} = \frac{X_{\bullet\bullet\bullet}}{IJK} \tag{1.137}$$

$$X_{i\bullet\bullet}^2 = \left(\sum_j \sum_k X_{ijk} \right)^2 \tag{1.138}$$

Presentation of definitions and procedures for the three-way analysis of variance, no replications of the design points L=1, is given in Table 1.26. For this kind of analysis with replicating L>1, the calculation is given in Table 1.27. The convention on notations were followed here:

$$X_{\bullet\bullet\bullet\bullet} = \sum_i \sum_j \sum_k \sum_l X_{ijkl}; \quad \bar{X}_{\bullet\bullet\bullet\bullet} = \frac{X_{\bullet\bullet\bullet\bullet}}{IJKL}; \quad X_{i\bullet\bullet\bullet}^2 = \left(\sum_j \sum_k \sum_l X_{ijkl} \right)^2 \tag{1.139}$$

Example 1.31 [13]

The following results were obtained in a two-factor, two-level experiment involving a study of the effect of temperature, time on the percentage yield of a reaction:

	Temperature [°C]			
Time	110	115	120	$X_{i\bullet\bullet}$
1h	5; 6	9; 7	10; 11	48.0
2h	10.8	11; 12	13; 15	69.0
	$X_{\bullet j\bullet} = 29.0$	39.0	49.0	$X_{\bullet\bullet\bullet} = 117.0$
	$\sum\sum X_{\bullet jk}^2 = 225.0$	395.0	615.0	$\sum\sum\sum X_{ijk}^2 = 1235.0$

Table 1.25 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Temperature	2	50.00	25.00	20.00	$F_{2;6;0.95}$=5.14
Time	1	36.75	36.75	29.40	$F_{1;6;0.95}$=5.99
Interaction	2	0.00	0.00	0.00	$F_{2;6;0.95}$=5.14
Error	6	7.50	1.25	–	–
Total	11	94.25	–	–	–

$$SS_T = \sum_i \sum_j \sum_k X_{ijk}^2 - \frac{X_{\bullet\bullet\bullet}^2}{IJK} = 1235.0 - \frac{117.0^2}{2 \times 3 \times 2} = 94.25$$

$$SS_C = \frac{\sum_j X_{\bullet j\bullet}^2}{IK} - \frac{X_{\bullet\bullet\bullet}^2}{IJK} = \frac{29^2 + 39^2 + 49^2}{2 \times 2} - \frac{117.0^2}{2 \times 3 \times 2} = 50.0$$

Table 1.26 Three-way analysis of variance: without replications

Source of variation	Degrees of freedom	Sum of squares definition	Sum of squares calculation form	Mean square	Test statistic
Between columns	J-1	$SS_C = IK\sum_j \left(\bar{X}_{\bullet j\bullet} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_C = \frac{\sum_j X^2_{\bullet j\bullet}}{IK} - \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_C = \frac{SS_C}{J-1}$	$F_C = \frac{MS_C}{MS_E}$
Between rows	I-1	$SS_R = JK\sum_i \left(\bar{X}_{i\bullet\bullet} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_R = \frac{\sum_i X^2_{i\bullet\bullet}}{JK} - \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_R = \frac{SS_R}{I-1}$	$F_R = \frac{MS_R}{MS_E}$
Between layers	K-1	$SS_L = IJ\sum_k \left(\bar{X}_{\bullet\bullet k} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_L = \frac{\sum_k X^2_{\bullet\bullet k}}{IJ} - \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_L = \frac{SS_L}{K-1}$	$F_L = \frac{MS_L}{MS_E}$
Interaction C × R	(J-1)(I-1)	$SS_{CR} = K\sum_i\sum_j \left(\bar{X}_{ij\bullet} - \bar{X}_{i\bullet\bullet} - \bar{X}_{\bullet j\bullet} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_{CR} = \frac{\sum_i\sum_j X^2_{ij\bullet}}{K} - \frac{\sum_i X^2_{i\bullet\bullet}}{JK} - \frac{\sum_j X^2_{\bullet j\bullet}}{IK} + \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_{CR} = \frac{SS_{CR}}{(I-1)(J-1)}$	$F_{CR} = \frac{MS_{CR}}{MS_E}$
Interaction C × L	(J-1)(K-1)	$SS_{CL} = I\sum_j\sum_k \left(\bar{X}_{\bullet jk} - \bar{X}_{\bullet j\bullet} - \bar{X}_{\bullet\bullet k} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_{CL} = \frac{\sum_j\sum_k X^2_{\bullet jk}}{I} - \frac{\sum_j X^2_{\bullet j\bullet}}{IK} - \frac{\sum_k X^2_{\bullet\bullet k}}{IJ} + \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_{CL} = \frac{SS_{CL}}{(J-1)(K-1)}$	$F_{CL} = \frac{MS_{CL}}{MS_E}$
Interaction R × L	(I-1)(K-1)	$SS_{RL} = J\sum_i\sum_k \left(\bar{X}_{i\bullet k} - \bar{X}_{i\bullet\bullet} - \bar{X}_{\bullet\bullet k} - \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_{RL} = \frac{\sum_i\sum_k X^2_{i\bullet k}}{J} - \frac{\sum_i X^2_{i\bullet\bullet}}{JK} - \frac{\sum_k X^2_{\bullet\bullet k}}{IJ} + \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_{RL} = \frac{CC_{RL}}{(I-1)(K-1)}$	$F_{RL} = \frac{MS_{RL}}{MS_E}$
Interaction C × R × L	(I-1)(J-1)(K-1)	$SS_{CRL} = \sum_i\sum_j\sum_k \left(X_{ijk} - \bar{X}_{ij\bullet} - \bar{X}_{i\bullet k} - \bar{X}_{\bullet jk} + \right.$ $\left. + \bar{X}_{i\bullet\bullet} + \bar{X}_{\bullet j\bullet} + \bar{X}_{\bullet\bullet k} + \bar{X}_{\bullet\bullet\bullet}\right)^2$	$SS_{CRL} = \sum_i\sum_j\sum_k X^2_{ijk} - \frac{\sum_i\sum_j X^2_{ij\bullet}}{K} - \frac{\sum_i\sum_k X^2_{i\bullet k}}{J} -$ $\frac{\sum_j\sum_k X^2_{\bullet jk}}{I} + \frac{\sum_i X^2_{i\bullet\bullet}}{JK} + \frac{\sum_j X^2_{\bullet j\bullet}}{IK} + \frac{\sum_k X^2_{\bullet\bullet k}}{IJ} - \frac{X^2_{\bullet\bullet\bullet}}{IJK}$	$MS_{CRL} = \frac{SS_{CRL}}{(I-1)(J-1)(K-1)}$	$F_{CRL} = \frac{MS_{CRL}}{MS_E}$

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Table 1.27 Three-way analysis of variance: with replications

Source of variation	Degrees of freedom	Sum of squares definition	Mean square	Test statistic
Between columns	J-1	$SS_C = \frac{\sum_j X^2_{\bullet j \bullet \bullet}}{IKL} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$	$MS_C = \frac{SS_C}{J-1}$	$F_C = \frac{MS_C}{MS_E}$
Between rows	I-1	$SS_R = \frac{\sum_i X^2_{i \bullet \bullet \bullet}}{JKL} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$	$MS_R = \frac{SS_R}{I-1}$	$F_R = \frac{MS_R}{MS_E}$
Between layers	K-1	$SS_L = \frac{\sum_k X^2_{\bullet\bullet k \bullet}}{IJL} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$	$MS_L = \frac{SS_L}{K-1}$	$F_L = \frac{MS_L}{MS_E}$
Interaction C × R	(I-1)(J-1)	$SS_{CR} = \frac{\sum_i \sum_j X^2_{ij\bullet\bullet}}{KL} - \frac{\sum_i X^2_{i\bullet\bullet\bullet}}{JKL} - \frac{\sum_j X^2_{\bullet j \bullet \bullet}}{IKL} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$	$MS_{CR} = \frac{SS_{CR}}{(I-1)(J-1)}$	$F_{CR} = \frac{MS_{CR}}{MS_E}$
Interaction C × L	(J-1)(K-1)	$SS_{CL} = \frac{\sum_j \sum_k X^2_{\bullet jk\bullet}}{IL} - \frac{\sum_j X^2_{\bullet j \bullet \bullet}}{IKL} - \frac{\sum_k X^2_{\bullet\bullet k \bullet}}{IJL} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$	$MS_{CL} = \frac{SS_{CL}}{(J-1)(K-1)}$	$F_{CL} = \frac{MS_{CL}}{MS_E}$

Table 1.27 Continued

Source of variation	Degrees of freedom	Sum of squares definition	Mean square	Test statistic
Interaction R × L	(I-1)(K-1)	$SS_{RL} = \frac{\sum_i \sum_k X_{i\bullet k\bullet}^2}{JL} - \frac{\sum_i X_{i\bullet\bullet\bullet}^2}{JKL} - \frac{\sum_k X_{\bullet\bullet k\bullet}^2}{IJL} + \frac{X_{\bullet\bullet\bullet\bullet}^2}{N}$	$MS_{RL} = \frac{SS_{RL}}{(I-1)(K-1)}$	$F_{RL} = \frac{MS_{RL}}{MS_E}$
Interaction C × R × L	(I-1)(J-1)(K-1)	$SS_{CRL} = \frac{\sum_i \sum_j \sum_k X_{ijk\bullet}^2}{L} - \frac{\sum_i \sum_j X_{ij\bullet\bullet}^2}{KL} - \frac{\sum_i \sum_k X_{i\bullet k\bullet}}{JL} - \frac{\sum_j \sum_k X_{\bullet jk\bullet}^2}{IL} + \frac{\sum_i X_{i\bullet\bullet\bullet}^2}{JKL} + \frac{\sum_j X_{\bullet j\bullet\bullet}^2}{IKL} + \frac{\sum_k X_{\bullet\bullet k\bullet}^2}{IJL} - \frac{X_{\bullet\bullet\bullet\bullet}^2}{N}$	$MS_{CRL} = \frac{SS_{CRL}}{(I-1)(J-1)(K-1)}$	$F_{CRL} = \frac{MS_{CRL}}{MS_E}$
Error	N-IJK	$SS_E = \sum_i \sum_j \sum_k \sum_l X_{ijkl}^2 - \frac{\sum_i \sum_j \sum_k X_{ijk\bullet}^2}{L}$	$MS_E \frac{SS_E}{N-IJK}$	–
Total	N-1	$SS_T = \sum_i \sum_j \sum_k \sum_l X_{ijkl}^2 - \frac{X_{\bullet\bullet\bullet\bullet}^2}{N}$	–	–

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$$SS_R = \frac{\sum_i X_{i\bullet\bullet}^2}{JK} - \frac{X_{\bullet\bullet\bullet}^2}{IJK} = \frac{48^2 + 69^2}{3 \times 2} - \frac{117.0^2}{2 \times 3 \times 2} = 36.75$$

$$SS_{CR} = \frac{\sum_i \sum_j X_{ij\bullet}^2}{K} - \frac{\sum_i X_{i\bullet\bullet}^2}{JK} - \frac{\sum_j X_{\bullet j\bullet}^2}{IK} + \frac{X_{\bullet\bullet\bullet}^2}{IJK} =$$

$$= \frac{11^2+18^2+\ldots+21^2+28^2}{2} - \frac{48^2+69^2}{3\times2} - \frac{29^2+39^2+49^2}{2\times2} + \frac{117.0^2}{2\times3\times2} = 0.0$$

$$SS_E = SS_T - SS_C - SS_R - SS_{CR} = 94.25 - 50.0 - 36.75 + 0.0 = 7.50$$

The results clearly show that temperature and time at 95% and 99% confidence level respectively have a significant effect on production yield. It is also clear that there is no interaction between temperature and time factors. Experimental error is small with respect to interaction so that the used measurement equipment can be considered satisfactory.

Example 1.32 [12]

The influence of glass types and coatings on specific conductivity was tested in developing cathode tubes for TV sets. From each obtained value 260mA were subtracted so that the coded values are:

	Type of coating			
Type of glass	A	B	C	$X_{i\bullet\bullet)}$
1	4; 6; 5	8; 10; 7	2; 5; 6	53.0
2	-6;-5;-4	0;-4;-5	-8;-7;-6	-45.0
	$X_{\bullet j\bullet}$ = 0.0	16.0	-8.0	$X_{\bullet\bullet\bullet}$ = 8.0
	$\sum\sum X_{\bullet jk}^2 = 154,0$	254.0	214.0	$\sum\sum\sum X_{ijk}^2 = 622,0$

I=2;J=3;K=3

$$SS_T = 622.0 - \frac{8.0^2}{2\times3\times3} = 618.44$$

$$SS_C = \frac{0.0^2+16.0^2+(-8)^2}{2\times3} - \frac{8.0^2}{2\times3\times3} = 53.33 - 3.56 = 49.77$$

$$SS_R = \frac{53^2+(-45)^2}{3\times3} - \frac{8.0^2}{2\times3\times3} = 537.11 - 3.56 = 533.55$$

$$SS_{CR} = \frac{15^2+(-15)^2+\ldots+13^2+(-21)^2}{3} - 537.11 - 53.33 + 3.56 = 1.79$$

SS_E=618.44-49.77-533.55-1.79=33.33

Table 1.28 Analysis of variance

Source of variation	f	SS	MS	F	$F_{T.}$
Type of coating	2	49.77	24.88	8.95	$F_{2;12;0.95}$=3.89
Type of glass	1	533.55	533.55	191.92	$F_{1;12;0.95}$=4.75
Interaction	2	1.79	0.89	0.32	$F_{2;12;0.95}$=3.89
Error	12	33.33	2.78	–	–
Total	17	618.44	–	–	–

The analysis of variance clearly shows that glass type has a dominant influence on cathode tube conductivity. The effect of coating type is also significant, while interaction is of no importance.

Example 1.33 [10]

A group of 24 mice was randomly divided into six subgroups of four and each mouse got an insulin shot. Taking into account three levels of insulin doses and two procedures of preparing insulin, A and B, reduction of sugar per cent in the blood of mice was measured some time after injecting insulin. The obtained results are shown in the following table:

Table 1.29 Analysis of variance

		Doses			$X_{i\bullet\bullet}$
		2.29	3.63	5.57	
Preparation	A	17	64	62	636
		21	48	72	
		49	34	61	
		54	63	91	
		33	41	56	
	B	37	64	62	576
		40	34	57	
		16	64	72	
$X_{\bullet j\bullet}$		267	412	533	$1212 = X_{\bullet\bullet\bullet}$
$\sum_j \sum_k X^2_{\bullet jk}$		10361	22554	36443	69358

SS_T=69358-61206=8152 ; SS_C=65640.25-61206=4434.25
SS_R=61356-61206=150.00 ; SS_{CR}=188275-65640.25-61356+61206=72.75
SS_E=8152-4434.25-150-72.75=3495

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Table 1.30 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Doses	2	4434.25	2217.125	11.419	$F_{2;18;0.95}$=3.55
Preparation	1	150.00	150.000	0.773	$F_{1;18;0.95}$=4.41
Interaction	2	72.75	36.375	0.187	$F_{2;18;0.95}$=3.55
Error	18	3495.00	194.167	–	–
Total	23	8152.00	–	–	–

Thus, we can with 95% confidence level say that reduction of sugar percent in the blood of mice is significantly influenced only by the size of insulin dose.

Example 1.34 [15]
The following results were obtained in a three-way, two-level experiment where temperature A, pressure B and catalyst C effects on chemical reaction yield were analyzed:

Table 1.31 Analysis of variance

	A_1		A_2		$X_{i\bullet\bullet}$
	C_1	C_2	C_1	C_2	
B_1	58	64	71	78	271
B_2	53	63	69	81	266
$\sum X_{\bullet jk}$	111	127	140	159	$X_{\bullet\bullet\bullet} = 537$
$\sum_j \sum_k X^2_{\bullet jk}$	6173	8065	9802	12645	$\sum_i \sum_j \sum_k X^2_{ijk} = 36685$

$$X_{1\bullet\bullet} = 58 + 64 + 71 + 78 = 271; \quad X_{2\bullet\bullet} = 53 + 63 + 69 + 81 = 266;$$
$$X_{\bullet\bullet\bullet} = 537; X^2_{\bullet\bullet\bullet} = 288369$$

$$\sum_i X^2_{i\bullet\bullet} = 271^2 + 266^2 = 144197; \quad \sum_i \sum_j \sum_k X^2_{ijk} = 36685$$

$$\sum_j X^2_{\bullet j\bullet} = (58 + 64 + 53 + 63)^2 + (71 + 78 + 69 + 81)^2 = 146045$$

$$\sum_k X^2_{\bullet\bullet k} = (58 + 71 + 53 + 69)^2 + (64 + 63 + 78 + 81)^2 = 144797$$

$$\sum_i \sum_j X^2_{ij\bullet} = (58 + 64)^2 + (71 + 78)^2 + (53 + 63)^2 + (69 + 81)^2 = 73041$$

$$\sum_i \sum_k X^2_{i\bullet k} = (58 + 71)^2 + (68 + 78)^2 + (53 + 69)^2 + (63 + 81)^2 = 72425$$

$$\sum_j \sum_k X^2_{\bullet jk} = (58 + 53)^2 + (64 + 63)^2 + (71 + 69)^2 + (78 + 81)^2 = 73331$$

$$SS_C = \frac{\sum_j X_{\bullet j \bullet}^2}{IK} - \frac{X_{\bullet\bullet\bullet}^2}{IJK} = \frac{146045}{2\times2} - \frac{288369}{2\times2\times2} = 465.1$$

$$SS_R = \frac{\sum_i X_{i\bullet\bullet}^2}{JK} - \frac{X_{\bullet\bullet\bullet}^2}{IJK} = \frac{144197}{2\times2} - \frac{288369}{2\times2\times2} = 3.1$$

$$SS_L = \frac{\sum_k X_{\bullet\bullet k}^2}{IJ} - \frac{X_{\bullet\bullet\bullet}^2}{IJK} = \frac{144797}{2\times2} - \frac{288369}{2\times2\times2} = 153.1$$

$$SS_{CR} = \frac{\sum_i \sum_j X_{ij\bullet}^2}{K} - \frac{\sum_i X_{i\bullet\bullet}^2}{JK} - \frac{\sum_j X_{\bullet j\bullet}^2}{IK} + \frac{X_{\bullet\bullet\bullet}^2}{IJK}$$
$$= \frac{73041}{2} - \frac{144197}{2\times2} - \frac{146045}{2\times2} + \frac{288369}{2\times2\times2} = 6.1$$

$$SS_{CL} = \frac{\sum_j \sum_k X_{\bullet jk}^2}{I} - \frac{\sum_j X_{\bullet j\bullet}^2}{IK} - \frac{\sum_k X_{\bullet\bullet k}^2}{IJ} + \frac{X_{\bullet\bullet\bullet}^2}{IJK}$$
$$= \frac{73331}{2} - \frac{146045}{2\times2} - \frac{144797}{2\times2} + \frac{288369}{2\times2\times2} = 1.1$$

$$SS_{RL} = \frac{\sum_i \sum_k X_{i\bullet k}^2}{J} - \frac{\sum_i X_{i\bullet\bullet}^2}{JK} - \frac{\sum_k X_{\bullet\bullet k}^2}{IJ} + \frac{X_{\bullet\bullet\bullet}^2}{IJK}$$
$$= \frac{72425}{2} - \frac{144197}{2\times2} - \frac{144797}{2\times2} + \frac{288369}{2\times2\times2} = 10.1$$

$$SS_{CRL} = \sum_i \sum_j \sum_k X_{ijk}^2 - \frac{\sum_i \sum_j X_{ij\bullet}^2}{K} - \frac{\sum_j \sum_k X_{\bullet jk}^2}{I} - \frac{\sum_i \sum_k X_{i\bullet k}^2}{J}$$
$$+ \frac{\sum_i X_{i\bullet\bullet}^2}{JK} + \frac{\sum_j X_{\bullet j\bullet}^2}{IK} + \frac{\sum_k X_{\bullet\bullet k}^2}{IJ} - \frac{X_{\bullet\bullet\bullet}^2}{IJK} =$$
$$= 36685 - \frac{73041}{2} - \frac{73331}{2} - \frac{72425}{2} + \frac{144197}{2\times2} + \frac{146045}{2\times2} + \frac{144797}{2\times2} - \frac{288369}{2\times2\times2} = 0.125$$

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This is an example of three-way analysis of variance with no design-point replication. As we have only one value for each set of factors, the variance or the mean square within the cell as an estimate of system variance cannot be calculated. In the lack of error variance, or rather *Reproducibility variance*. *Interaction* of a higher order can be used as error estimate for the F-test. Although all statisticians do not agree with this approach, the three-way interaction variance C × R × L was taken as the error estimate for F-test. The tabular results show that only the effects of columns and layers, or temperature and catalyst, are significant. Pressure and interaction are not important at the 95% confidence level. The other approach in estimating repro-

ducibility variance is to join several interactions. We can assume that all four interac-

Table 1.32 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Between columns	1	465.1	465.1	3720	$F_{1;1;0.95}$=161
Between rows	1	3.1	3.1	24.8	$F_{1;1;0.95}$=161
Between layers	1	153.1	153.1	1223	$F_{1;1;0.95}$=161
Interaction C × R	1	6.1	6.1	48.8	$F_{1;1;0.95}$=161
Interaction C × L	1	1.1	1.1	8.0	$F_{1;1;0.95}$=161
Interaction R × L	1	10.1	10.1	80	$F_{1;1;0.95}$=161
Interaction C × R × L	1	0.125	0.125	–	–

tions are unimportant, i.e. we can join their degrees of freedom and sums of squares to obtain the estimate of variance error. In that case the degree of freedom is k=4 and the sum of squares 6.1+1.1+10.1+0.125=17.425; so that MS_E=4.28. With the $F_{1;4;0.95}$=7.71 value, the mean square for any effect must exceed the value $4.28 \times 7.71 = 33.0$; so that the effect could be statistically important at 95% confidence level. This proves again that in this case only the effects of temperature and catalyst are statistically significant.

Example 1.35 [14]

Transfer of mass from liquid into solid particles of an expanded fountain-fluidized bed is developed in the annulus and fountain according to the following criterion equation

$$Sh = A \times \mathrm{Re}_A^a \times \mathrm{Re}_S^b \times \left(\frac{H}{D}\right)^c$$

where:

Sh is *Sherwood's*-criterion;

Re_A and Re_S- *Reynold's* Number – criteria for annulus and fountain, and

H/D-height simplex of the added active coal bed versus column diameter.

By varying Re_A, Re_S and H/D, we obtained the values of *Sherwood's*-criterion as shown in the table. By applying analysis of variance, verify the given criterion equation by establishing the effects of the observed factors on the basis of experimental values.

Table 1.33 Analysis of variance

	Re_{S1}		Re_{S2}		$X_{i\bullet\bullet\bullet}$
	Re_{A1}	Re_{A2}	Re_{A1}	Re_{A2}	
$(H_0/D)_1$	2.023	2.235	2.110	2.123	16.958
	2.024	2.212	2.112	2.119	
	[4.047]	[4.447]	[4.222]	[4.242]	
$(H_0/D)_2$	1.790	1.897	1.966	1.983	15.265
	1.782	1.868	1.988	1.991	
	[3.572]	[3.765]	[3.954]	[3.974]	
$X_{\bullet jk\bullet}$	7.619	8.212	8.176	8.216	$X_{\bullet\bullet\bullet\bullet} = 32.223$
$\sum_j \sum_k \sum_l X^2_{\bullet jkl}$	14.569	16.976	16.730	16.894	65.169

I=2; J=2; K=2; L=2; N=IJKL=16 ;

$$\sum_i \sum_j \sum_k \sum_l X^2_{ijkl} = 65.169; \quad X_{\bullet\bullet\bullet\bullet} = 32.223; \quad X^2_{\bullet\bullet\bullet\bullet} = 1038.322;$$

$$\sum_i \sum_j \sum_k X^2_{ijk\bullet} = 130.335; \quad \sum_i X^2_{i\bullet\bullet\bullet} = 16.958^2 + 15.265^2 = 520.594;$$

$$\sum_j X^2_{\bullet j\bullet\bullet} = (7.619 + 8.212)^2 + (8.176 + 8.216)^2 = 519.319$$

$$\sum_k X^2_{\bullet\bullet k\bullet} = (7.619 + 8.176)^2 + (8.212 + 8.216)^2 = 519.361$$

$$\sum_i \sum_j X^2_{ij\bullet\bullet} = (4.047 + 4.447)^2 + (4.222 + 4.242)^2 + (3.572 + 3.765)^2 + (3.954 + 3.974)^2 = 260.472$$

$$\sum_i \sum_k X^2_{i\bullet k\bullet} = (4.047 + 4.222)^2 + (3.572 + 3.954)^2 + (4.447 + 4.242)^2 + (3.765 + 3.974)^2 = 260.408$$

$$\sum_j \sum_k X^2_{\bullet jk\bullet} = (4.047 + 3.572)^2 + (4.447 + 3.765)^2 + (4.222 + 3.954)^2 + (4.242 + 3.974)^2 = 259.836$$

$$SS_T = \sum_i \sum_j \sum_k \sum_l X^2_{ijkl} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = 65.169 - \frac{32.223^2}{16} = 0.274$$

$$SS_L = \frac{\sum_k X^2_{\bullet\bullet k\bullet}}{IJL} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = \frac{519.361}{2\times2\times2} - 64.895 = 0.025;$$

$$SS_C = \frac{\sum_j X^2_{\bullet j\bullet\bullet}}{IKL} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = \frac{519.319}{2\times2\times2} - \frac{32.223^2}{16} = 0.020$$

$$SS_R = \frac{\sum_i X^2_{i\bullet\bullet\bullet}}{JKL} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = \frac{520.594}{2\times2\times2} - 64.895 = 0.179$$

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$$SS_{CR} = \frac{\sum_i \sum_j X^2_{ij\bullet\bullet}}{KL} - \frac{\sum_i X^2_{i\bullet\bullet\bullet}}{JKL} - \frac{\sum_j X^2_{\bullet j\bullet\bullet}}{IKL} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$$
$$= \frac{260.472}{2\times2} - \frac{520.594}{2\times2\times2} - \frac{519.319}{2\times2\times2} + 64.895 = 0.024$$

$$SS_{CL} = \frac{\sum_j \sum_k X^2_{\bullet jk\bullet}}{IL} - \frac{\sum_j X^2_{\bullet j\bullet\bullet}}{IKL} - \frac{\sum_k X^2_{\bullet\bullet k\bullet}}{IJL} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$$
$$= \frac{259.836}{2\times2} - \frac{519.319}{2\times2\times2} - \frac{519.361}{2\times2\times2} + 64.895 = 0.019$$

$$SS_{RL} = \frac{\sum_i \sum_k X^2_{i\bullet k\bullet}}{JL} - \frac{\sum_i X^2_{i\bullet\bullet\bullet}}{JKL} - \frac{\sum_k X^2_{\bullet\bullet k\bullet}}{IJL} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$$
$$= \frac{260.408}{2\times2} - \frac{520.594}{2\times2\times2} - \frac{519.361}{2\times2\times2} + 64.895 = 0.003$$

$$SS_E = \sum_i \sum_j \sum_k \sum_l X^2_{ijkl} - \frac{\sum_i \sum_j \sum_k X^2_{ijk\bullet}}{L} = 65.169 - \frac{130.335}{2} = 0.001$$

SS_{CRL}=SS_T-SS_C-SS_R-SS_L-SS_{CR}-SS_{CL}-SS_{RL}-SS_E
=0.274-0.020-0.179-0.025-0.024-0.019-0.003-0.001=0.003

Table 1.34 Analysis of variance

Source of variation	F	SS	MS	F	F_T
Re_s of fountain	1	0.020	0.020	166.67	$F_{1;8;0.95}$=5.32
H_0D simplex	1	0.179	0.179	1491.67	$F_{1;8;0.95}$=5.32
Re_A annulus	1	0.025	0.025	208.33	$F_{1;8;0.95}$=5.32
$Re_S \times H_0/D$	1	0.024	0.024	200.00	$F_{1;8;0.95}$=5.32
$Re_S \times Re_A$	1	0.019	0.019	158.33	$F_{1;8;0.95}$=5.32
$H_0/D \times Re_a$	1	0.003	0.003	25.000	$F_{1;8;0.95}$=5.32
$Re_S \times H_0/D \times Re_a$	1	0.003	0.003	25.000	$F_{1;8;0.95}$=5.32
Error	8	0.001	0.00012	–	–
Total	15	0.274	–	–	–

It is of interest for variance methodology that complex values such as non dimensional Reynold's numbers and simplex were used as factors in this example. Analysis of variance has shown that all factors and interactions are important, which verifies the assumed criterion equation. The low error value of the experiment characterizes good experiment reproducibility and simultaneously indicates the fact that all essential factors have been taken into consideration.

Example 1.36 [4]

The table below gives the results of an experiment as *niacin*-contents in peeled and processed peas of three granulations (A, B and C), after three kinds of preparations (R_1, R_2 and R_3).

Table 1.35 Peeled peas

A			B			C		
R_1	R_2	R_3	R_1	R_2	R_3	R_1	R_2	R_3
65	90	44	59	70	83	88	80	123
87	94	92	63	65	95	60	81	95
48	86	88	81	78	88	96	105	100
28	70	75	80	85	99	87	130	131
20	78	80	76	74	81	68	122	121
22	65	70	85	73	98	75	130	115
24	75	73	64	61	95	96	121	127
47	98	88	96	71	95	98	125	99
28	95	77	91	63	90	84	172	101
42	66	72	65	54	76	82	133	111
411	**817**	**759**	**760**	**694**	**900**	**834**	**1199**	**1123**

Table 1.36 Processed peas

A			B			C		
R_1	R_2	R_3	R_1	R_2	R_3	R_1	R_2	R_3
62	106	126	150	138	150	146	52	100
113	107	193	112	120	112	172	97	133
171	79	122	136	135	126	138	112	125
135	122	115	120	126	123	124	116	124
123	125	126	118	120	125	113	121	115
132	96	110	134	132	110	121	99	122
120	111	98	125	135	125	125	120	112
117	116	115	114	124	120	116	121	116
153	124	112	112	137	110	165	122	99
132	126	109	120	125	125	137	134	105
1258	**1112**	**1226**	**1241**	**1292**	**1226**	**1357**	**1094**	**1151**

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These tables give:

I=2; J=3; K=3; L=10; N=I × J × K × L=180. ; $X_{\bullet\bullet\bullet\bullet} = 18454$ $X^2_{\bullet\bullet\bullet\bullet} = 340\,550\,116$

$$\sum_i \sum_j \sum_k \sum_l X^2_{ijkl} = 65^2 + 87^2 + 48^2 + \ldots + 116^2 + 99^2 + 105^2 = 22054828;$$

Table 1.37 Peeled Peas

	A	B	C
R_1	1258	1241	1357
R_2	1112	1292	1094
R_3	1226	1226	1151
Sum	3596	3759	3602

Table 1.38 Processed Peas

	A	B	C
R_1	411	760	834
R_2	817	694	1199
R_3	759	900	1123
Sum	1987	2354	3156

By compiling the given sums, we can make up the following three tables:

Table 1.39 Accessory Table

	Peeled	Processed	Sum
A	1987	3596	5583
B	2354	3759	6113
C	3156	3602	6758
Sum	7497	10957	

Table 1.40 Accessory Table

	Peeled	Processed
R_1	2005	3856
R_2	2710	3498
R_3	2782	3603

Table 1.41 Accessory Table

	A	B	C	Sum
R_1	1669	2001	2191	5861
R_2	1929	1986	2293	6208
R_3	1985	2126	2274	6385

The following arithmetic is necessary for analysis of variance:

$$\sum_i X_{i\bullet\bullet}^2 = 7497^2 + 10957^2 = 176\,260\,858\,;$$

$$\sum_j X_{\bullet j\bullet}^2 = 5583^2 + 6113^2 + 6758^2 = 114\,209\,222$$

$$\sum_k X_{\bullet\bullet k}^2 = 5861^2 + 6208^2 + 6385^2 = 113\,658\,810$$

$$\sum_i \sum_j X_{ij\bullet}^2 = 1987^2 + 2354^2 + 3156^2 + 3596^2 + 3759^2 + 3602^2 = 59\,485\,522$$

$$\sum_i \sum_k X_{i\bullet k}^2 = 2005^2 + 2710^2 + 2782^2 + 3856^2 + 3498^2 + 3603^2 = 59\,189\,998$$

$$\sum_j \sum_k X_{\bullet jk}^2 = 1669^2 + 1929^2 + 1985^2 + 2001^2 + 1986^2 + 2126^2 + 2191^2 + 2293^2 + 2274^2 = 38\,144\,306$$

$$\sum_i \sum_j \sum_k X_{ijk}^2 = 411^2 + 760^2 + 834^2 + 817^2 + 694^2 + 1199^2 + 759^2 + 900^2 + 1123^2 + 1258^2 + +1241^2 + 1357^2 + 1112^2 + 1292^2 + 1094^2 + 1226^2 + 1226^2 + 1151^2 = 20\,073\,704$$

SS_T=2 054 828-1 891 945=162 883 ; SS_C=1 958 454-1 891 945=66 509
SS_R=1 903 487-1 891 945=11 542 , SS_I=1 894 314-1 891 945=2 369
SS_{CR}=1 982 851-1 958 454-1 903 487+1 891 945=12 855
SS_{CI}=1 973 000-1 958 454-1 894 314+1 891 945=12 177
SS_{RI}=1 907 215-1 903 487-1 894 314+1 891 945=1 359 ; SS_E=2 054 828-2 007 370=47 458
SS_{CRI}=2007370-1982851-1973000-1907215+1958454+1903487+1894314-1891945=8614

Table 1.42 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Between columns	1	66509	66509.00	227.03	$F_{1;162;0.95}$=3.84
Between rows	2	11542	5771.00	19.70	$F_{2;162;0.95}$=3.00
Between layers	2	2369	1184.50	4.04	$F_{2;162;0.95}$=3.00
Interaction C × R	2	12855	6427.50	21.94	$F_{2;162;0.95}$=3.00
Interaction C × L	2	12177	6088.50	20.78	$F_{2;162;0.95}$=3.00
Interaction R × L	4	1359	339.75	1.16	$F_{4;162;0.95}$=2.37
Interaction C × R × L	4	8614	2153.5	7.35	$F_{4;162;0.95}$=2.37
Error	162	47458	292.95	–	–
Total	179	162883	909.96	–	–

The table clearly shows that all factors and interactions are statistically significant at 95% confidence level except for the second-order interaction R × L. It should be noted that columns refer to granulometric contents of peas, sorts to peeled and processed peas, and layers to preparation kinds (R_1, R_2 and R_3).

Example 1.37 [17]

Analysis of variance has also been applied in testing three factors of composite rocket propellant burning rate in *Crawford's*-bomb at 100bar and 20 °C temperature. The analyzed factors are: contents of fine fraction in bimodal oxidizer mixture (C); contents ratio of oxidizer mixture-aluminum powder (B) and contents of burning rate catalyst (A). The obtained burning rate values are given in the table in mm/s:

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Table 1.43 Analysis of variance

	A_1		A_2		$\sum$
	C_1	C_2	C_1	C_2	
	7.1	7.5	9.8	15.6	
B_1	7.0	7.0	9.6	15.9	79.5
	[14.1]	[14.5]	[19.4]	[31.5]	
	7.8	9.1	10.6	20.0	
B_2	7.8	9.4	11.1	20.8	96.6
	[15.6]	[18.5]	[21.7]	[40.8]	
	29.7	33.0	41.1	72.3	176.1

I=2; J=2; K=2; L=2; N=I × J × K × L=16 ;

$$\sum_i \sum_j \sum_k \sum_l X_{ijkl}^2 = 2250.09; \quad X_{\bullet\bullet\bullet\bullet} = 176.1; \quad X_{\bullet\bullet\bullet\bullet}^2 = 31011.21$$

$$\sum_i X_{i\bullet\bullet\bullet}^2 = 79.5^2 + 96.6^2 = 15651.81$$

$$\sum_j X_{\bullet j\bullet\bullet}^2 = (29.7 + 33.0)^2 + (41.1 + 72.3)^2 = 16790.85$$

$$\sum_k X^2_{\bullet\bullet k\bullet} = (29.7+41.1)^2+(33.0+72.3)^2 = 16\,100.73$$

$$\sum_i \sum_j X^2_{ij\bullet\bullet} = (14.1+14.5)^2+(19.4+31.5)^2+(15.6+18.5)^2+(21.7+40.8)^2 = 8\,477.83$$

$$\sum_i \sum_k X^2_{i\bullet k\bullet} = (14.1+19.4)^2+(15.6+21.7)^2+(14.5+31.5)^2+(18.5+40.8)^2 = 8\,146.03$$

$$\sum_j \sum_k X^2_{\bullet jk\bullet} = (14.1+15.6)^2+(14.5+18.5)^2+(19.4+21.7)^2+(31.5+40.8)^2 = 8\,887.59$$

$$\sum_i \sum_j \sum_k X^2_{ijk\bullet} = 14.1^2+14.5^2+19.4^2+31.5^2+15.6^2+18.5^2+21.7^2+40.8^2 = 4\,498.81$$

$$SS_T = \sum_i \sum_j \sum_k \sum_l X^2_{ijkl} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = 2250.09 - \frac{31\,011.21}{16} = 311.89$$

$$SS_C = \frac{\sum_j X^2_{\bullet j\bullet\bullet}}{I\times K\times L} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = \frac{16\,790.85}{8} - \frac{31\,011.21}{16} = 160.66$$

$$SS_R = \frac{\sum_i X^2_{i\bullet\bullet\bullet}}{J\times K\times L} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = \frac{15\,651.81}{8} - \frac{31\,011.21}{16} = 18.28$$

$$SS_L = \frac{\sum_k X^2_{\bullet\bullet k\bullet}}{I\times J\times L} - \frac{X^2_{\bullet\bullet\bullet\bullet}}{N} = \frac{16\,100.73}{8} - \frac{31\,011.21}{16} = 74.39$$

$$SS_{CR} = \frac{\sum_i \sum_j X^2_{ij\bullet\bullet}}{K\times L} - \frac{\sum_i X^2_{i\bullet\bullet\bullet}}{J\times K\times L} - \frac{\sum_j X^2_{\bullet j\bullet\bullet}}{I\times K\times L} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$$
$$= \frac{8477.83}{2\times 2} - \frac{15651.81}{2\times 2\times 2} - \frac{16790.85}{2\times 2\times 2} + \frac{31011.21}{16} = 2.33$$

$$SS_{CL} = \frac{\sum_j \sum_k X^2_{\bullet jk\bullet}}{I\times L} - \frac{\sum_j X^2_{\bullet j\bullet\bullet}}{I\times K\times L} - \frac{\sum_k X^2_{\bullet\bullet k\bullet}}{I\times J\times L} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$$
$$= \frac{8887.59}{2\times 2} - \frac{16790.85}{2\times 2\times 2} - \frac{16100.73}{2\times 2\times 2} + \frac{31011.21}{16} = 48.65$$

$$SS_{RL} = \frac{\sum_i \sum_k X^2_{i\bullet k\bullet}}{J\times L} - \frac{\sum_i X^2_{i\bullet\bullet\bullet}}{J\times K\times L} - \frac{\sum_k X^2_{\bullet\bullet k\bullet}}{I\times J\times L} + \frac{X^2_{\bullet\bullet\bullet\bullet}}{N}$$
$$= \frac{8146.03}{2\times 2} - \frac{15651.81}{2\times 2\times 2} - \frac{16100.73}{2\times 2\times 2} + \frac{31011.21}{16} = 5.64$$

$$SS_E = \sum_i \sum_j \sum_k \sum_l X_{ijkl}^2 - \frac{\sum_i \sum_j \sum_k X_{ijk\bullet}^2}{L} = 2250.09 - \frac{4498.81}{2} = 0.69$$

$$SS_{CRL} = SS_T - SS_C - SS_R - SS_L - SS_{CR} - SS_{CL} - SS_{RL} - SS_E$$
$$= 311.89–160.66–18.28–74.39–2.33–48.65–5.64–0.69 = 1.25$$

Table 1.44 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Factor A	1	160.66	160.66	1868.14	$F_{1;8;0.95}$=5.32
Factor B	1	18.28	18.28	212.56	$F_{1;8;0.95}$=5.32
Factor C	1	74.39	74.39	865.00	$F_{1;8;0.95}$=5.32
Interaction A × B	1	2.33	2.33	27.09	$F_{1;8;0.95}$=5.32
Interaction A × C	1	48.65	48.65	565.70	$F_{1;8;0.95}$=5.32
Interaction B × C	1	5.64	5.64	65.58	$F_{1;8;0.95}$=5.32
Interaction A × B × C	1	1.25	1.25	14.53	$F_{1;8;0.95}$=5.32
Error	8	0.69	0.086	–	–
Total	15	311.89	–	–	–

The mentioned analysis of variance clearly shows that the most important influence (the top F-criterion calculated value) on the burning rate of composite rocket propellant comes with catalyst content change. One can also notice the high F-criterion value of the interaction A × C, or the interactions of catalyst and fine fraction contents in the oxidizer mixture. As the significance of second-order interactions means that the power of influence of one factor on response depends on the level the other factor is at, we can conclude that the efficiency of the burning-rate catalyst does not depend only on its mass but also on granulometric contents of the oxidizer. Small experimental error indicates that its reproducibility is very good and that, in general, all factors influencing the composite rocket propellant burning rate have been taken into consideration.

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Problem 1.26 [9]

The following three process factors were tested to determine the influence of conditions in the technology of isatin production:

- factor A-basic raw material concentration;
- factor B-time of reaction;
- factor C-temperature of reaction.

Table 1.45 Analysis of variance

	C_1		C_2	
	B_1	B_2	B_1	B_2
A_1	6.08	6.53	6.79	6.73
	6.31	6.12	6.77	6.49
A_2	6.04	6.43	6.68	6.08
	6.09	6.36	6.38	6.23

The results are given in the table. By applying analysis of variance, select the factors according to effects of their influence on the measured chemical reaction yield.

Problem 1.27 [4]

The following data give the yields of a product that resulted from trying catalysts from four different suppliers in a process. Determine:

	Catalyst					
I	36	33	35	34	32	34
II	35	37	36	35	37	36
III	35	39	37	38	39	38
IV	34	31	35	32	34	33

a) Are yields influenced by catalysts?
b) What are your recommendations in the selection of a catalyst?

Problem 1.28 [4]

Random samples of size ten were drawn from normal populations A, B, C and D. The measurements of property X are given in the table below along with totals and sums of squares in columns.

Are there significant differences between the population means?

Table 1.46 Analysis of variance

	X_A	X_B	X_C	X_D	
	3.355	0.273	3.539	3.074	
	1.086	2.155	2.929	3.103	
	2.367	1.725	3.025	2.389	
	0.248	0.949	4.097	4.766	
	1.694	0.458	2.236	2.553	
	1.546	1.455	3.256	3.821	
	1.266	2.289	3.374	1.905	
	0.713	2.673	1.781	2.350	
	0.000	1.800	2.566	1.161	
	3.406	2.407	2.510	2.122	
$\sum X_{i\bullet}$	15.681	16.184	29.313	27.244	$X_{\bullet\bullet}$
$\bar{X}$	1.5681	1.6184	2.9313	2.7244	
$\sum_j X_{ij}^2$	37.071327	32.339668	90.081121	83.620342	$\sum_i \sum_j X_{ij}^2$

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Problem 1.29

Samples of steels from four different batches were analyzed for carbon content. The results are shown below for quadruplicate determinations by the same analyst. Are the carbon contents (given in weight per cent) of these batches the same? What are the 99% confidence limits on the average carbon content of each?

Table 1.47 Analysis of variance

	No1	No2	No3	No4	
	0.39	0.36	0.32	0.43	
	0.41	0.35	0.36	0.39	
	0.36	0.35	0.42	0.38	
	0.38	0.37	0.40	0.41	
$X_{\bullet j}$	1.54	1.43	1.50	1.61	$X_{\bullet\bullet} = 6.08$
$\bar{X}_{\bullet j}$	0.3850	0.3575	0.3750	0.4025	
$\sum_j X_{\bullet j}^2$	0.5942	0.5115	0.5684	0.6495	$\sum_i \sum_j X_{ij}^2 = 2.3236$

■ **Problem 1.30**

The torque outputs for several pneumatic actuators at several different supply pressures are given in the table below.

a) Do the different supply pressures significantly affect the output?
b) Does the output vary for the different models?

Table 1.48 Analysis of variance

	P_1	P_2	P_3
A	205	270	340
B	515	700	880
C	1775	2450	3100
D	7200	9600	12100

■ **Problem 1.31 [4]**

Results of a laboratory analysis for the specific rate constant for the saponification of ethyl acetate by NaOH at 0 [°C] are given below. One of the runs was performed with an electric stirrer, while the others were hand stirred. The results were analyzed by the differential and integral methods. Does the electric stirrer make a significant difference to the results? Does the method of analysis of data make a significant difference?

Table 1.49 Analysis of variance

	With stirrer		**Without stirrer**	
Integral	0.02058	0.02121	0.01849	0.01816
Method	0.02214	0.02073	0.01951	0.01884
Differential	0.01995	0.02003	0.01725	0.01752
Method	0.01968	0.01982	0.01696	0.01726

■ **Problem 1.32 [12]**

Observe an experiment aimed at defining the factors that have an essential effect on energy consumption in processing metal on a lathe with ceramic cutting tools. In metal processing, in fact, the energy is measured by a dynamometer and it is proportional to power consumption. Some of the factors influencing the observed response are: type of cutting tools, angle of cutting, depth of cutting, speed of moving the metal, type of cutting, and turning speed of axis. In this experiment the cutting depth was fixed at 2.5mm, speed of moving the metal at 0.3mm/min, and turning speed was fixed at 1000min^{-1}.

The other three factors were varied on three levels: type of cutting tools, angle of cutting and type of cutting. The experiment data after four replications are given in the table:

Table 1.50 Analysis of variance

Type of cutting	Cutting tools T_1		Cutting tools T_2	
	Cutting angle 15(B_1)	Cutting angle 30($B_{2)}$	Cutting angle 15(B_1)	Cutting angle 30($B_{2)}$
Continuously	29.0	28.5	28.0	29.5
C_1	26.5	28.5	28.5	32.0
	30.5	30.0	28.0	29.0
	27.0	32.5	25.0	28.0
With interruption	28.0	27.0	24.5	27.5
C_2	25.0	29.0	25.0	28.0
	26.5	27.5	28.0	27.0
	26.5	27.5	26.0	26.0

■ **Problem 1.33 [10]**

In developing a procedure for bacteriological testing of milk, samples were tested in an apparatus that includes two components: bottles and kivets. All six combinations of two bottle types and three kivet types were tested ten times for each sample. The table contains data on the number of positive tests in each of ten testings. If we remember section 1.1.1 then the obtained values of positive tests are a random variable with the binomial distribution. For a correct application of the analysis of variance procedure, the results should be normally distributed. It is therefore possible to transform the obtained results by means of arcsine mathematical transformation for the purpose of example of three-way analysis of variance with no replications, no such transformations are necessary. The experiment results are given in the table:

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Table 1.51 Analysis of variance

Bottles	I			II		
Kivets	A	B	C	A	B	C
Milk samples	1	1	1	1	3	2
	3	4	2	2	1	3
	3	2	4	3	3	6
	2	4	1	1	0	0
	2	1	3	2	4	6
	1	1	2	0	2	1
	5	5	5	3	5	5
	1	1	1	0	2	0
	0	1	2	2	2	2
	3	4	5	1	1	3
	0	0	4	0	2	1
	0	1	2	0	3	1

Problem 1.34 [21]

Comparative analysis of different technological solutions for producing nitrate compounds was done in lab conditions. Three factors of importance for nitrate compounds production yield were tested:

a) Time of dosing the nitric acid-A;
b) Time of mixing the reaction mixture-B;
c) The factor of mixture remains from the previous batch-C.

The experimental results are given in the table:

Table 1.52 Analysis of variance

	C_1		C_2	
	A_1	A_2	A_1	A_2
B_1	87.2	88.4	86.7	89.2
B_2	82.0	83.0	83.4	83.7

Problem 1.35 [9]

Paste is produced in a batch-type technological procedure. Three samples (A, B, and C) were taken from each batch and each of them tested twice for tearing strength. The obtained results and their sums are in the table. Do the analysis of variance.

Table 1.53 Analysis of variance

Batch	Sample A			Sample B			Sample C			Sum	Mean
			Sum			Sum			Sum		
1	50.3	49.8	100.1	50.1	49.5	99.6	51.1	49.4	100.5	300.2	50.03
2	45.8	45.4	91.2	44.4	44.7	89.1	44.7	44.4	89.1	269.4	44.90
3	41.0	41.4	82.4	42.7	41.6	84.3	43.1	43.3	86.4	253.1	42.18
4	48.7	50.0	98.7	48.0	50.4	98.4	47.9	48.5	96.4	293.5	48.92
5	48.9	49.4	98.3	48.4	46.8	95.2	46.5	45.4	91.9	285.4	47.57
6	47.0	46.1	93.1	47.4	47.2	94.6	45.1	47.5	92.6	280.3	46.72
7	46.3	45.0	91.3	44.6	44.0	88.6	45.6	44.2	89.8	269.7	44.95
8	44.9	42.3	87.2	45.1	43.4	88.5	43.3	41.6	84.9	260.6	43.43
9	55.7	55.4	111.1	56.3	56.3	112.6	55.1	55.0	110.1	333.8	55.63
Sum			853.4			850.9			841.7	2546.0	–
Mean			47.4			47.3			46.8	47.1	–

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Problem 1.36 [9]

The table shows logarithms of $CaCO_3$-chalk filling weights from two deposits A and B as determined in 11 laboratories with three replications. Use analysis of variance to determine whether there are significant differences between labs or chalk deposits.

Table 1.54 Analysis of variance

Laboratory	Chalk A				Chalk B				Mean
				Mean				Mean	
1	0.851	0.851	0.851	0.851	0.681	0.686	0.681	0.683	0.767
2	0.863	0.866	0.860	0.863	0.690	0.690	0.695	0.692	0.777
3	0.854	0.854	0.854	0.854	0.686	0.686	0.690	0.687	0.771
4	0.863	0.869	0.869	0.867	0.690	0.690	0.690	0.690	0.779
5	0.869	0.872	0.872	0.871	0.699	0.703	0.699	0.700	0.786
6	0.875	0.869	0.872	0.872	0.686	0.690	0.686	0.687	0.780
7	0.485	0.857	0.851	0.851	0.672	0.672	0.681	0.675	0.763
8	0.869	0.869	0.872	0.870	0.699	0.695	0.695	0.696	0.783
9	0.857	0.857	0.857	0.857	0.681	0.681	0.681	0.681	0.769
10	0.869	0.881	0.875	0.875	0.695	0.690	0.690	0.692	0.783
11	0.881	0.881	0.881	0.881	0.708	0.708	0.708	0.708	0.795
Mean				0.865				0.690	0.777

■ **Problem 1.37 [9]**

Effects on physical properties of vulcanized rubber were analyzed in a research lab. Based on research results the influence of three factors on the strength of vulcanized rubber was tested:

a) five qualities of rubber filler- factor A;
b) three methods of previous rubber treatment-factor B;
c) four qualities of raw rubber-factor C.

Do the analysis of variance.

Table 1.55 Analysis of variance

	Factor C											
	Factor B			**Factor B**			**Factor B**			**Factor B**		
Factor A	404	478	530	381	429	528	316	376	390	423	482	550
	392	418	431	239	251	249	186	207	194	410	416	452
	348	381	460	327	372	482	290	315	350	383	376	496
	296	291	333	165	232	242	158	279	220	301	306	330
	186	198	125	129	157	197	105	163	190	213	200	255

■ **Problem 1.38 [9]**

In a chemical plant nine aluminum alloys were tested for their resistance to corrosion. Four locations were chosen inside the chemical plant for this experiment and at each such place one plate of all nine alloys was placed. Exposure to chemical corrosion lasted one year. After the experiment four researchers examined the plates randomly and gave a mark from 1 to 10 to each plate depending on the observed resistance to corrosion. The experiment was aimed at asserting which of the offered aluminum alloys had the best resistance to corrosion at one or at all locations of the chemical plant. It was also interesting to see how much the researchers agreed or disagreed in their estimates of resistance to corrosion. Thus, the experiment had included three factors: nine aluminum alloys, four locations and four researchers. Experimental results are shown in the following table:

Table 1.56 Analysis of variance

Location	Researcher	Alloys									Sum
		I	II	III	IV	V	VI	VII	VIII	IX	
I	A	5	5	5	4	6	6	1	6	7	45
	B	4	5	5	4	5	3	1	5	7	39
	C	7	7	7	7	8	5	4	7	7	59
	D	6	5	4	5	7	6	3	6	7	49
	Sum	22	22	21	20	26	20	9	24	28	192
II	A	8	7	7	7	5	4	5	4	5	52
	B	7	8	6	7	6	5	3	7	8	57
	C	9	9	9	9	8	6	7	8	8	73
	D	8	8	7	7	5	5	7	4	5	56
	Sum	32	32	29	30	24	20	22	23	26	238
III	A	4	4	5	3	4	3	0	5	5	33
	B	1	3	3	2	5	2	0	4	5	25
	C	5	5	5	6	6	4	3	7	9	150
	D	3	3	7	2	3	3	1	6	6	34
	Sum	13	15	20	13	18	12	4	22	25	142
IV	A	6	5	6	5	6	4	4	7	5	48
	B	1	3	6	5	5	4	3	6	5	38
	C	5	5	7	6	8	7	5	8	8	59
	D	5	3	5	3	5	3	3	7	6	40
	Sum	17	16	24	19	24	18	15	28	24	185
Grand mean		84	85	94	82	92	70	50	97	103	757

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Problem 1.39 [4]

The following values show the percentage of conversion of vinegar acid into anhydride of vinegar acid at 750 °C by catalytic cracking at different percentages of triethyl phosphate (TEP) as catalyst. The input flow of material was changeable.

a) Does the catalyst percentage affect conversion?
b) Does the input flow of material affect conversion?

Table 1.57 Analysis of variance

Flow GAL/l	Level of catalyst		
	0.5% TEF	0.3% TEF	0.1% TEF
200.0	77.85	76.03	73.87
147.0	89.12	88.94	87.65
98.5	99.09	97.14	91.78
61.0	99.55	99.51	97.60

Problem 1.40 [4]

In an air-water contact utilizing a packed tower, the water-air contact is occurring whereby the gas film heat transfer coefficient depends on the flow of liquid and gaseous phases. By varying these two flows, coefficient values of heat transfer were obtained and they are presented in the table:

Table 1.58 Analysis of variance

Gas flow G	Liquid flow L			
	190	**250**	**300**	**400**
200	200	226	240	261
400	278	312	330	381
700	369	416	462	517
1100	500	575	645	733

a) Perform the indicated two-way AHOVA for these data. Use the α=0.01 significance level.

b) Which variable has the greater effect on the heat transfer coefficient?

Problem 1.41 [18]

This problem presents the results of researching strength at tear of adhesive systems on surfaces with no primers and those where primers were applied. A distinction was also made as for thickness of the sample. Values of strength at tear with an asterix were obtained from samples of one thickness and those without an asterix from samples of different thickness.

Apply the two-way analysis of variance to values with an asterix so as to determine the significance of effects of different adhesive systems and previous preparation of surface.

Table 1.59 Analysis of variance

	Adhesive systems					Adhesive systems			
	I	II	III	IV		I	II	III	IV
W	60.0*	57.0*	19.8*	52.0*	W	59.0*	51.0*	29.4*	49.0*
i	73.0	52.0	32.0	77.0	i	78.0	52.0	37.8	77.0
t	63.0*	52.0*	19.5*	53.0*	t	48.0*	44.0*	32.2*	59.0*
h	79.0	56.0	33.0	78.0	h	72.0	42.0	36.7	76.0
	57.0*	55.0*	19.7*	44.0*		51.0*	42.0*	37.1*	55.0*
P	70.0	57.0	32.0	70.0	n	72.0	51.0	35.4	79.0
r	53.0*	59.0*	21.6*	48.0*	o	49.0*	54.0*	31.5*	54.0*
i	69.0	58.0	34.0	74.0		75.0	47.0	40.2	78.0
m	56.0*	56.0*	21.1*	48.0*	p	45.0*	47.0*	31.3*	49.0*
e	78.0	52.0	31.0	74.0	r	71.0	57.0	40.7	79.0
r	57.0*	54.0*	19.3*	53.0*	i	48.0*	56.0*	33.0*	58.0*
	74.0	53.0	27.3	81.0	m.	72.0	45.0	42.6	79.0

Problem 1.42

Repeat Problem 1.41 but for values with no asterix.

Problem 1.43

As the values with an asterix in Problem 1.41 mean that they were obtained for one thickness of the sample and those with no asterix for another one, apply the three-way analysis of variance for all the data in Problem 1.41 taking into account the new sample thickness factor.

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Problem 1.44

In a pilot-plant for producing composite rocket propellants 12 batches were produced, by which seven experimental rocket motors were cast and cured for each batch. By static firing of these motors at 25 °C burning-rate laws as a function of pressure were obtained. From such a law a rate value at 70bar pressure was obtained. The propellant batches were mixed with two batches of ammonium perchlorate and two types of catalysts. Besides, each batch was repeated three times. The burning-rate data at 25 °C and P=70bar for all mixing conditions of the propellants are given in the table below. By applying the two-way analysis of variance with replication, determine with 95% confidence level whether the catalyst type and preparation of 10 μm of ammonium perchlorate essentially affect the propellant burning rate at 25 °C and 70bar?

Table 1.60 Analysis of variance

	Catalyst A			Catalyst B		
Batch AP (A)	14.199	14.197	14.193	15.716	15.612	15.682
Batch AP (B)	14.398	14.418	14.307	15.616	15.912	15.482

Summary of analysis of variance

Analysis of variance is a procedure by which the total variance is divided into sources of variations. Depending on the experiment design done, it is possible to separate from the total variance a different number of sources of variations. However, no matter how many sources of variations were selected, they all refer both to those that occur under the influence of systematic variations and to the error resulting from random variations. The aim of applying the analysis of variance method is to answer the question: is the difference between the obtained response means for the tested factors a result of the influence of tested factors or has it occurred randomly.

In solving the problem we shall start from the null hypothesis, which assumes that the response means for the tested factors are the same and that the existing differences have occurred randomly.

Hence the null hypothesis is:

$$H_0 : \bar{X}_1 = \bar{X}_2 = ... = \bar{X}_i$$

The answer to the question is obtained by either accepting or rejecting the null hypothesis, achieved through the analysis of variance method and performed in the following way:

- If the obtained differences of the response means for the varied factors are significant, the null hypothesis is rejected and we conclude that they result from the influence of the factor.
- If the obtained differences are not important, we accept the null hypothesis and consider the existing differences as random ones.

Practically, this is what it looks like:

If the calculated value of the applied statistical test is lower than the associated tabular value for the threshold or level of significance α, i.e. the confidence is $(1-\alpha) \times 100\%$,the influence or effect of the factor is not statistically significant. The hypothesis is accepted and we conclude that the differences between the means are a consequence of random fluctuation factors.

If the calculated value of the applied statistical test is above the associated tabular value for the threshold or level of significance α, i.e. the confidence is $(1-\alpha) \times 100\%$, the difference or effect is statistically significant. The hypothesis is rejected and the conclusion is that such a difference may in the future be expected in $(1-\alpha) \times 100\%$ cases provided the experiment is done under identical conditions. Hence only in $\alpha \times 100\%$ cases can a different outcome be expected.

To enable successful application of analysis of variance, certain assumptions that are in the basis of models and conclusions drawn should be fulfilled. One of the

basis assumptions is additivity of effects of some tests and model components in general. The next assumption is that ε_{ij} error has a normal distribution for each test individually and for the experiment as a whole. The third one refers to the statistical equality or homogeneity of test variances. The given assumptions justify the use of the F-test.

Barttlet's test for equality of variances

The analysis of variance technique for testing equality of means is a rather *robust* procedure. That is, when the assumption of normality and homogeneity of variances is "slightly" violated the F-test remains a good procedure to use. In the one-way model, for example, with an equal number of observations per column it has been exhibited that the F-test is not significantly effected. However, if the sample size varies across columns, then the validity of the F-test can be greatly affected. There are various techniques for testing the equality of k variances $\sigma_1^2, \sigma_2^2, \ldots, \sigma_k^2$. We discuss here the most widely used technique, *Barttlet's* χ^2 test for homogeneity of variances. Let $S_1^2, S_2^2, \ldots, S_k^2$, be k independent sample variances corresponding to k normal populations with means μ_i and variance σ_i^2, (i=1.2,...,k). Suppose n_1-1, n_2-1,...,n_k-1, are the respective degrees of freedom. Barttlet [19] proposed the statistic:

$$\chi^2 = \left[(\ln V)\sum_{i=1}^{k}(n_i-1) - \sum_{i=1}^{k}(n_i-1)\ln S_i^2\right] \Big/ \ell \tag{1.140}$$

where:

$$V^2 = \left[\sum_{i=1}^{k}(n_i-1)S_i^2\right] \Big/ \left[\sum_{i=1}^{k}(n_i-1)\right] \tag{1.141}$$

and ln denotes the natural logarithm. The denominator in Eq. (1.140) is defined by:

$$\ell = 1 + \frac{1}{3(k-1)}\left[\sum_{i=1}^{k}\frac{1}{n_i-1} - 1 \Big/ \sum_{i=1}^{k}(n_i-1)\right] \tag{1.142}$$

It can be shown that the statistic in Eq. (1.140) has an approximate χ^2-distribution with k-1 degrees of freedom when used as a test statistic for $H_0 : \sigma_1^2 = \ldots = \sigma_k^2$. Given k random samples of sizes n_1, n_2,...,n_k, from k independent normal populations the statistic χ^2 in (1.140) can be used to test H_0. Recall that a sample variance is:

$$S^2 = \left(\sum_{i=1}^{n} X_i^2 - n\bar{X}^2\right) \Big/ (n-1) \tag{1.143}$$

If all the samples are of the same size, say n, then Barttlet's statistic in Eq. (1.140) becomes:

$$\chi^2 = (n-1)\left[\left(k\ln V - \sum_{i=1}^{k}\ln S_i^2\right)/\ell\right] \tag{1.144}$$

where:

$$\ell = 1 + (1+k)/[3k(n-1)]$$

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The connection between natural and decade logarithms gives:

$$\chi^2 = 2.3026(n-1)\left(k\log V - \sum_{i=1}^{k}\log S_i^2\right)\Big/\ell \tag{1.45}$$

If n_i=n, and i=1, 2,..., k, then:

$$V = \left(\sum_{i=1}^{k} S_i^2\right)\Big/k \tag{1.46}$$

The rejection region for testing H_0: $\sigma_1^2 = \sigma_2^2 = \ldots = \sigma_k^2$ is $\chi^2 \succ \chi^2_{(k-1),1-\alpha}$.

Example 1.38 [4]

In an experiment to determine the effects of sample size and amount of liquid phase on the height equivalent to a theoretical plate (HETP) in gas chromatography, it was necessary to utilize solid support material from different batches. It was therefore imperative that the resulting data be checked for homogeneity prior to attempting to develop any quantitative expressions regarding the effects of these variables on HETP. Several sets of data points were selected at random and examined using Bartlett's test.

In particular, a set of four HETP values obtained for cyclohexane for a 4 μl sample injected into a 40% β, β′-oxydipropionitrile column were:

X_1=0.44; $X_1^2 = 0.1936$
X_2=0.44; $X_2^2 = 0.1936$
X_3=0.40; $X_3^2 = 0.1600$
X_4=0.43; $X_4^2 = 0.1849$

$$\sum X_i = 1.71; \quad \sum X_i^2 = 0.7321 \quad ; \quad \bar{X} = \frac{1.71}{4} = 0.4275; \quad \bar{X}^2 = 0.18275625$$

$$S^2 = \frac{\sum_{i=1}^{n} X_i^2 - n\bar{X}^2}{n-1} = \frac{0.7321 - 4\times 0.18275625}{3} = 3.583\times 10^{-4}$$

The variance of 10 cyclohexane data sets, each consisting of four observations, are thus calculated and presented below. In this case n_i=4; and i=1, 2,...,10 and k=10.

Liquid phase, β, β′-oxydipropionitrile	S_i^2	$\log S_i^2$
40%-4 ml sample	0.0003583	-3.44575
30%-8 ml sample	0.0002250	-3.64782
20%-10 ml sample	0.0002250	-3.64782
20%-4 ml sample	0.0000916	-4.03810
10%-4 ml sample	0.0000916	-4.03810
5%-10 ml sample	0.0003000	-3.52288
5%-2 ml sample	0.0002250	-3.64782
3%-8 ml sample	0.0002250	-3.64782
3%-6 ml sample	0.0003000	-3.52288
10%-2 ml sample	0.0002250	-3.64782
	0.0022665	-36.80681

The computations needed to calculate χ^2 according to Eq. (1.145) are:

V=0.0022665/10=0.00022665 logV=-3.64464
k logV=10 (-3.64464)=-36.4464
ℓ = 1+11/30 × 3=1.1222
χ^2=2.3026 × (4-1)[-36.4464-(-36.80681)]/1.1222=2.218

The tabular value $\chi^2_{9;0.975}$=19.0 is considerably higher than the arithmetic so that with a 95% probability one can assert that the variances are equal. A simpler and more frequently used test for equality of variances is *Cochran's* test.

Cochran's test for equality of variances

Cochran's test on equality of variances is relatively simple and its arithmetic is brought down to calculating:

$$C = \frac{S_i^2 \max}{\sum_i S_i^2} \tag{1.147}$$

where:

S_i^2 max is the largest variance in a sequence of tested variances,

$\sum_i S_i^2$-the sum of all test variances in the experiment.

If the arithmetic of *Cochran's* test is below the tabular value, the null hypothesis that variances are equal is accepted. Tabular values are in Table I and are determined for the associated threshold or significance level α=0.05 and degree of freedom: f_1=n-1 number of data used in calculating the variance S_i^2 is reduced by one, or in other words the number of replicated design points (trials) is reduced by one and the f_2-number of variances or number of trials.

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Example 1.39

Apply *Cochran's* test for equality of variances to Example 1.38.

The previous example gives:

S_i^2 max=0.0003583 ; $\sum_i S_i^2$=0.0022665 ; f_1=n-1=4-1=3 ; f_2=10 ; α=0.05.

C=0.000383/0.0022665=0.1689 ; $C_{3;10;0.95}$=0.3733

Due to the fact that the calculated value is smaller than the tabular value, the null hypothesis that variances are equal is accepted.

Data transformation

By transformation, the original experimental data obtain other values on the basis of which the analysis of variance is done. There are three main reasons for its application:

- to achieve equality of variances;
- to achieve normality of data distribution;
- to make the test effects additive.

F-distribution is influenced only by test additivity which has been mentioned as the third reason for data transformation. It is good that all three assumptions of analysis of variance are achieved by one transformation. Generally speaking, transformation will be applied always when there is a link between the test means in the experiment and their variances. Due to such a general condition, one should find the corresponding transformation. We shall first indicate the cases when no form of transformation could be applied:

- The test averages are approximately equal while their variances are heterogeneous.
- The test averages are independent of their variances.
- Variances are equal but distributions are heterogeneous.

In such a case the solution could be to apply one of the nonparametric statistical procedures. Even in this case, nonparametric statistics is characterized by less rigorous assumptions than those for parametric one, and it is relatively less efficient. There are different forms of transformation. We shall present here the most widely applied ones in research studies.

Transformation based on square root from data $X'=\sqrt{X}$ is applied when the test values and variances are proportional as in *Poisson's* distribution. If the data come from counting up and the number of units is below 10 transformation form $X'=\sqrt{X+1}$ and \text $X'=\sqrt{X}+\sqrt{X+1}$ is used. If the test averages and their standard deviations are approximately proportional, we use the logarithm transformation X′=log X. If there are data with low values or they have a zero value, we use X′=log (X+1). When the squares of arithmetical averages and standard deviations are proportional we use the reciprocal transformation: X′=1/X or X′=1/(X+1) if the values are small or are equal to zero. The transformation arc sin $\sqrt{X}$ is used when values are given as proportions and when the distribution is Binomial. If the test value of the experiment is zero then instead of it we take the value 1/(4n), and when it is 1, 1-1/(4n) is taken as the value and n is the number of values. Transforming values where the proportion varies between 0.30 and 0.70 is practically senseless. This transformation is done by means of special tables suited for the purpose.

Dilemmas often occur in actual cases in spite of such clear indications on which transformation to apply. There exist several formal procedures that may be of great help. A relatively simple procedure will be presented.

The calculation consists of taking in different transformations at each trial the highest and lowest values and establishing the range from their difference. Then the ratio between the largest and smallest ranges is established. The smallest ratio in the given transformation indicates the transformation form to be applied. As an illustration we shall take the experiment [20] with one factor where the influence of herbicides on weeds is researched and where the factor is varied on six levels and each test replicated five times.

Table 1.61 Herbicide influence on weeds in sugar beet

Number of replications	Factor levels						Sum
	A	B	C	D	E	F	
1	520	268	162	120	142	35	1247
2	530	270	164	121	149	30	1264
3	530	280	152	126	140	35	1263
4	490	269	148	111	141	24	1183
5	457	278	158	120	149	25	1187
Sum	2527	1365	784	598	721	149	6144

Table 1.62 Transformation on basis of largest and smallest trial values

	Factor						Largest and smallest value ratio
	A	B	C	D	E	F	
Largest value (V)	530	280	164	126	149	35	
Smallest value (M)	457	268	148	111	140	24	
Interval	73	12	16	15	9	11	73/9=8.11
$\sqrt{V}$	23.02	16.73	12.81	11.22	12.21	5.92	
$\sqrt{M}$	21.38	16.37	12.17	10.54	11.83	4.90	
Interval	1.64	0.36	0.64	0.68	0.38	1.02	1.64/0.36=4.55
log V	2.7243	2.4472	2.2148	2.1004	2.1732	1.5441	
log M	2.6599	2.4281	2.1703	2.0453	2.1461	1.3802	
Interval	0.0644	0.0191	0.0445	0.0551	0.0271	0.1639	0.1639/0.0191=8.58
1/V	0.00186	0.00357	0.00609	0.00794	0.00671	0.02857	
1/M	0.00219	0.00373	0.00676	0.00901	0.00714	0.04167	
Interval	0.00033	0.00016	0.00067	0.00107	0.00043	0.01310	0.01310/0.00016=81.88
$1/\sqrt{V}$	0.04344	0.05977	0.07806	0.08913	0.08190	0.16892	
$1/\sqrt{M}$	0.04677	0.06109	0.08217	0.09488	0.08453	0.20408	
Interval	0.00333	0.00132	0.00411	0.00575	0.00263	0.03516	0.03516/0.00132=26.64

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This procedure indicates that the transformation is the square root from data and is the most suitable for results of the experiment the aim of which was to find out the influence of herbicides on weeds in sugar beet. Before and after the application of transformation the normality of the data distribution should be checked.

Check of the null hypothesis on normality of data distribution

If the distribution of data or random value is unknown we can make up a *histogram* from them. Intervals associated with groups of random values within the same ranges are drawn on the abscissa. A rectangle the height of which is equal to the frequency of results appearing within the interval is drawn above each range, where n_g in n_g/N is the number of values within the observed range. The following algorithm for drawing up the histogram may be suggested:

- The region of random value changes X_{min}-X_{max} is divided into ε ranges. The number of ε ranges is determined by the formula:

$$\varepsilon=1+3.2 \log N \tag{1.148}$$

where:
N is the number of data in a sample.
The range width is calculated from equation:

$$\Delta g = \frac{X_{max}-X_{min}}{\varepsilon} \tag{1.149}$$

- The number of data n_g(g=1, 2,..., ε) within the range Δg or the frequency of values in the associated interval p_g is determined:

$$p_g = \frac{n_g}{N} \tag{1.150}$$

- Values falling within the g range have the characteristic mean X_g^*:

$$X_g^* = \frac{X_{g-1}-X_g}{2} \tag{1.151}$$

- Histogram $p_g \rightarrow X_{g\text{-}1}- X_g$.

After drawing up the histogram, the hypothesis on distribution normality of the obtained variation sequence is checked. The check is done by means of the criterion for estimating the difference between theoretical and empirical distributions. The most frequently used is Pirson's, criterion, determined by the formula:

$$\chi_R^2 = \frac{\sum_{g=1}^{\varepsilon}\left(n_g - N\widehat{p}_g\right)^2}{N\times\widehat{p}_g} \tag{1.152}$$

where:
ε is the number of ranges;
N is the number of values in a random sample;
N_g is the number of values within interval g;
$\widehat{p}_g$ is the probability of values falling within interval g as calculated by the theoretical distribution.

Pirson's criterion has f=ε-λ-1 degrees of freedom, where for a normal distribution λ=2. The $\widehat{p}_g$ probability is calculated by formula:

$$\widehat{p}_g = F_0\left(z_{g+1}\right) - F_0\left(z_g\right) \tag{1.153}$$

where:
z_g is the left g-interval limit, or:

$$z_g = \frac{X_g^*-\bar{X}}{S_X} \tag{1.154}$$

and F(z)-*Laplace's*-function:

$$F(z) = \frac{1}{\sqrt{2\pi}}\int_0^z e^{-X^2/2}dx \tag{1.155}$$

The values of the normed Laplace's-function are in Table J (appendix). When determining the given values z_g=z_{min} is replaced by $-\infty$, and z_g=z_{max} by $+\infty$. When the calculated value of the Pirson criterion is below the tabular value the null hypothesis on normal distribution of data is accepted:

$$\chi_R^2 \prec \chi_{TAB.}^2 \tag{1.156}$$

Example 1.40

In a chemical reactor 200 temperature values have been measured. The largest and smallest deviations from the average temperature were X_{max}=30 and X_{min}=-20. The values have been divided into 10 ranges. The accuracy of the measured temperatures was ±1 [°C]. By using Pirson's criterion with α=0.05 significance level, check the law on data distribution.

The mean and variance estimates are obtained from expressions:

$$\bar{X} = \sum_{g=1}^{10} X_g^* p_g = 4.30\,[°C] \quad ; \quad S_X^2 = \sum_{g=1}^{10} X_g^2 p_g - \bar{X} = 94.20[°C] \quad ; \quad S_X = 9.71\,[°C]$$

Table 1.63 Results of previous calculations

Number of ranges	Limits of ranges $X_g - X_{g+1}$	No. of values in the ragne n_g	Range center X_g^*	Relative frequency $p_g = n_g/N$
1	-20 – -15	7	-17.5	0.035
2	-15 –10	11	-12.5	0.055
3	-10 – -5	15	-7.5	0.075
4	-5 – 0	24	-2.5	0.120
5	0 – 5	49	2.5	0.245
6	5 – 10	41	7.5	0.205
7	10 – 15	26	12.5	0.130
8	15 – 20	17	17.5	0.085
9	20 – 25	7	22.5	0.035
10	25 – 30	3	27.5	0.015

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From Eq. (1.154) we can calculate the standardized and normal values of a random value, and then from Table J determine the $F_0(z_g)$ value keeping in mind that for z_g<0 $F_0(z_g)$=-$F_0(|z_g|)$. We can then form Table 1.64 by using (1.153) and (1.152).

Due to the small number of data in range g=10 we join it to range g=9. The number of degrees of freedom is therefore reduced by one. The arithmetic value for χ_R^2 is calculated from Eq. (1.152):

$$\chi_R^2 = 7.09; f = \varepsilon - \ell - 1 = 9 - 2 - 1 = 6$$

From Table D, $\chi_{TAB.}^2 = 12.6$ is obtained for f=6 and α=0.05, so that:

$$\chi_R^2 = 7.06 \prec \chi_{TAB.}^2 = 12.6$$

Table 1.64 Calculation results

g	z_g	$F_0(z_g)$	$\widehat{p}_g$	$N \times \widehat{p}_g$	$\frac{(n_g - N\times\widehat{p}_g)^2}{N\times\widehat{p}_g}$
1	$-\infty$	-0.5000	0.0239	4.78	1.04
2	-1.99	-0.4761	0.0469	9.38	0.28
3	-1.47	-0.4292	0.0977	19.54	1.05
4	-0.96	-0.3315	0.1615	32.30	2.13
5	-0.44	-0.1700	0.1979	39.58	2.24
6	0.07	0.0279	0.1945	38.90	0.11
7	0.59	0.2224	0.1419	28.38	0.20
8	1.10	0.3643	0.0831	16.62	0.01
9	1.62	0.4474	0.0526	10.52	0.03
10	2.13	0.4834	–	–	–
11	$+\infty$	–	–	–	–

Hence the hypothesis on normal distribution of measured temperature values in the chemical reactor is accepted.

Rejection of outliers

Outliers in a random sample often bring about wrong conclusions when testing and estimating parameters. Such values have to be rejected in a sample with great caution for this other extreme may affect the outcome of conclusions. The following procedure is suggested in reference [21]:

$$\Delta_{\max} = X_{\max} - \bar{X} \tag{1.157}$$

where:

X_{max} is the sample outlier.

Estimate the value:

$$|\Delta_{\max}| \succ C \times S_X \tag{1.158}$$

where:

C is the constant.

The outlier X_{max} is rejected from the sample if the inequality *(1.158)* is fulfilled. The procedure may be repeated several times whereby standard deviation S_X is determined from the remaining sample values each time. The constant C is determined from the t-Student's criterion by means of expression:

$$\left[\frac{N\times C^2\left(f+f_0-1\right)}{f\left(f+f_0-\frac{NC^2}{f}\right)}\right]^{0.5} \approx t_{\alpha=0.05}^{f_0+f-1} \tag{1.159}$$

where:

f=N-1; the degree of freedom of variance estimate S_X^2;

f_0 is any number of additional degrees of freedom (usually f_0=0).

Example 1.41

The following values of a product content have been measured by gas chromatography: X_1=23.2; X_2=23.4; X_3=23.5; X_4=24.1; X_5=25.5. Is X_5 the outlier and can it be rejected from this sample of values?

The calculated sample mean when X_5 is rejected is: $\bar{X}$=23.55.

$$\Delta_{\max} = \left|X_{\max} - \bar{X}\right| = |25.5 - 23.55| = 1.95\,; N = 5\,; n = N - 1 = 5 - 1 = 4$$

$$S_X^2 = \frac{\sum_{i=1}^{n}\left(X_i - \bar{X}\right)^2}{n-1} = \frac{\sum_{i=1}^{4}\left(X_i - 23.55\right)^2}{4-1} = 0.15\,;\; S_X = 0.39$$

For the significance level α=0.05; N=5; f=4 from Eq. (1.159) we get:

$$\left[\frac{5 \times C^2 \times 3}{4\frac{4-5C^2}{4}}\right]^{0.5} = 2.35$$

The result of this equation is C=1.44, so that in accord with Eq. (1.158) we get:

$|\Delta_{\max}| = 1.95 \succ C \times S_X = 1.44 \times 0.39 = 0.56$ and the hypothesis that X_5 is not the outlier is rejected.

Problem 1.45

From Table A of random numbers, 150 double digit numbers have been chosen. The data are in the next table. Check the normality of data distribution with 95% confidence level by using Pirson's criterion.

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Table 1.65 Random numbers

Number of intervals	Interval limits	Number of data in the range	Frequency
1	0 – 9	16	0.107
2	10 – 19	15	0.100
3	20 – 29	19	0.127
4	30 – 39	13	0.087
5	40 – 49	14	0.093
6	50 – 59	19	0.127
7	60 – 69	14	0.093
8	70 – 79	11	0.073
9	80 – 89	13	0.087
10	90 – 99	16	0.107

■ **Problem 1.46**

Data about average air temperatures for 320 days are given in the table. Check the normality of the temperature values by using *Pirson's* criterion with 5% significance level.

Table 1.66 Air temperatures

Number of intervals	Interval limits	Number of data in the range
1	-40 – -30	5
2	-30 – -20	11
3	-20 – -10	25
4	-10 – 0	42
5	0 – 10	88
6	10 – 20	81
7	20 – 30	36
8	30 – 40	20
9	40 – 50	8
10	50 – 60	4

■ **Problem 1.47**

The acidity of products of alcohol chlorinating has been measured by a pH meter. The following values have been obtained: 4.2; 4.4; 4.0; 4.2; 4.4; 4.6; 4.4; 4.6; 4.4; 5.2; 4.8; 4.5; 4.2. Is the value pH=5.2 the outlier?

■ **Problem 1.48**

Four batches of the same sort of composite rocket propellant have been cast in a lab. After curing, the experimental rocket motors were static fired at normal temperature. The following burning-rate values at P=70bar pressure have been obtained: 14.199 ; 14.531 ; 14.197 ; 14.193. Does the burning-rate value V=14.531mm/s represent the outlier with 95% confidence level?

1.6 Regression analysis

Regression is a highly useful statistical technique for developing a quantitative relationship between a dependent variable (response) and one or more independent variables (factors). It utilizes experimental data on the pertinent variables to develop a numerical relationship showing the influence of the independent variables on a dependent variable of the system.

Throughout engineering, regression may be applied to correlating data in a wide variety of problems ranging from the simple correlation of physical properties to the analysis of a complex industrial system. For example, in a catalytic reactor involving

a complex chemical reaction, regression methods have been used to develop an equation relating yield of desired product to entering concentrations, temperature, pressure, and residence time. In chemical engineering, regression may be applied to correlating data in a wide variety of problems, ranging from the simple correlation of physical properties to the analysis of a complex industrial reactor. If nothing is known from theory about the relationship among the pertinent variables, a function may be assumed and fitted to experimental data on the system.

Frequently a linear function is assumed. In other cases where a linear function does not fit the experimental data properly, the engineer might try a polynomial or exponential function. In a large number of cases theory produces incomplete models. Regression analysis is used in such cases for determining unknown coefficients in a theoretical equation from available experimental data. The theory of burning a rocket propellant, for instance, supposes that the linear burning rate changes, depending on pressure, in this way:

$$V=b\ p^n \tag{1.160}$$

In this case experimental data will be used for determining constants b and n by applying regression analysis.

1.6.1 Simple Linear Regression

In the simplest case the proposed functional relationship between two variables is:

$$Y=\beta_0+\beta_1 X+\varepsilon \tag{1.161}$$

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In this model Y is the dependent variable, X is the independent variable, and ε is a random error (or residual) which is the amount of variation in Y not accounted for by the linear relationship. The parameters β_0 and β_1 are called the regression coefficients that are unknown and are to be estimated. The variable X is not a random variable but takes on fixed values. It will be assumed that the errors ε are independent and have a normal distribution with mean 0 and variance σ^2, regardless of what fixed value of X is being considered. Taking the expectation of both sides of Eq. (1.161), we have:

$$E(Y)=\beta_0+\beta_1 X \tag{1.162}$$

where we note that the expected value of the errors is zero.

For a fixed value of X, the expectation in Eq. (1.162) is usually denoted by:

$$E(Y) = E(Y/X) = \mu_{Y/X}$$

Thus we can write:

$$E(Y) = E(Y/X) = \mu_{Y/X} = \beta_0 + \beta_1 X \tag{1.163}$$

Eq. (1.163) is called the regression of Y on X. The only random variables in Eq. (1.161) are Y and ε. Since the ε is normally distributed, the random variable Y has a normal distribution with mean $\mu_{Y/X}=\beta_0+\beta_1 X$ and variance σ^{2}. Geometric interpretation of the linear regression is shown in Fig. 1.19.

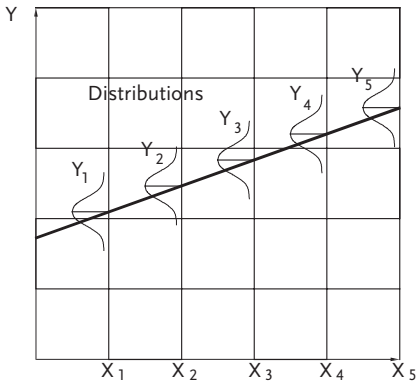

Figure 1.19 Geometric interpretation of linear regression

In order to estimate the relationship between Y and X suppose we have n observations on Y and X, denoted by (X_1,Y_1), (X_2,Y_2), ..., (X_n,Y_n). By Eqs.. (1.161) and (1.163) we can write the assumed relationship between Y and X as:

$$Y=E(Y/X)+\varepsilon \tag{1.164}$$

The objective here is to estimate β_0, and β_1 and thus E(Y/X) or Y in terms of the n observations. If b_0 and b_1 denote estimates of β_0 and β_1 then an estimate of E(Y) is denoted by $\widehat{Y} = \widehat{E}(Y) = b_0 + b_1 X$.

As mentioned before, one must differentiate population parameters and sample estimates of population parameters. The equation that describes population is given by expression (1.162). The population in this case is the basis for a hypothetical physical model that is correctly described by the given regression (1.162). We, however, take a data sample, or carry out the experiment presupposing the mathematical model set up is valid, and then using experimental results or the sample data, we calculate b_0 and b_1 as estimates of population parameters β_0 and β_1 . Thus each observed Y_i can be written as:

$$Y_i = \widehat{Y}_i + e_i,\ i = 1.2, ..., n \tag{1.165}$$

where:

$\widehat{Y}_i$ is the estimate Y_i,

e_i is error estimate ε_i .

The linear regression may be written as:

$$Y_i=\beta_0+\beta_1X_i+\varepsilon_i=b_0+b_1X_i+e_i,\ i=1.2,...,n \tag{1.166}$$

The Eq. (1.166) is illustrated in Fig. 1.20.

The point (X_i, Y_i) denotes the i-th observation. The "true" error or residual is $Y_i-(\beta_0+\beta_1X_i)$, the difference between the observed Y_i and the true unknown value $\beta_0+\beta_1X_i$. The observed residual e_i is $Y_i-(b_0+b_1X_i)=Y_i - \widehat{Y}_i$, which is the difference between the observed Y_i and the estimated $\widehat{Y}_i = b_0 + b_1 X_i$. The problem is now to obtain estimates b_0 and b_1 from the sample for the unknown parameters β_0 and β_1.

This can best be done by the *method of least squares*. This method minimizes the sum of squares of the differences between the predicted values and the experimental values for the dependent variable. This method minimizes the sum of square differences between estimates and experimental values of dependent variable-responses. The method is based on the principle that the best estimators of β_0 and β_1 are those that minimize the sum of squares due to error, SS_E.

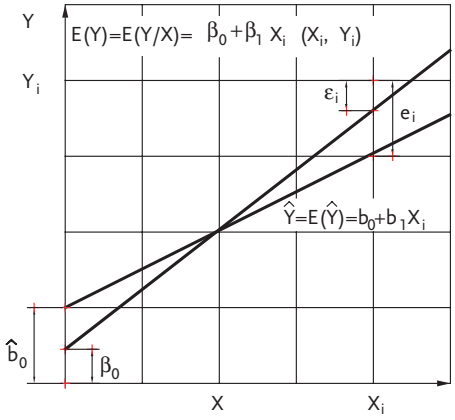

Figure 1.20 True and estimated regression lines

The error sum of squares is:

$$SS_E = \sum_{i=1}^{n} e_i^2 = \sum_{i=1}^{n} \left(Y_i - \hat{Y}_i\right)^2 \tag{1.167}$$

$$SS_E = \sum_{i=1}^{n} (Y_i - b_0 - b_1 X_i)^2 \tag{1.168}$$

To determine the minimum, the partial derivative of the error sum of squares with respect to each constant is set equal to zero to yield:

$$\frac{\partial(SS_E)}{\partial b_0} = \frac{\partial}{\partial b_0}\left[\sum_{i=1}^{n} (Y_i - b_0 - b_1 X_i)^2\right] = 0 \tag{1.169}$$

$$\frac{\partial(SS_E)}{\partial b_1} = \frac{\partial}{\partial b_1}\left[\sum_{i=1}^{n} (Y_i - b_0 - b_1 X_i)^2\right] = 0 \tag{1.170}$$

These equations (1.169) and (1.170) are called normal equations. Carrying out the differentiation, we obtain:

$$nb_0 + b_1 \sum_i X_i = \sum_i Y_i \tag{1.171}$$

$$b_0 \sum_i X_i + b_1 \sum_i X_i^2 = \sum_i X_i Y_i \tag{1.172}$$

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where all the summations go from i=1 to i=n. The solutions to these normal equations are:

$$b_0 = \bar{Y} - b_1\bar{X} \tag{1.173}$$

$$b_1 = \frac{\sum_i (X_i - \bar{X})(Y_i - \bar{Y})}{\sum_i (X_i - \bar{X})^2} \tag{1.174}$$

The estimator b_1 can also be written in the form:

$$b_1 = \frac{\sum_i X_i Y_i - n\bar{X}\bar{Y}}{\sum_i X_i^2 - n\bar{X}^2} \tag{1.175}$$

The error sum of squares can be written as:

$$SS_E = \sum_i (Y_i - \bar{Y})^2 - b_1^2 \sum_i (X_i - \bar{X})^2 \tag{1.176}$$

The first term on the right-hand side of Eq. (1.176) is the total corrected sum of squares SS_{TC}, of the Ys. The linear relationship between X and Y accounts for a reduction of $b_1^2 \sum (X_i - \bar{X})^2$ in SS_{TC}. That is, if there is no linear relationship between X and Y, β_1=0, then $\sum (Y_i - \bar{Y})^2 = SS_E$. If there is a linear relationship between X and Y, then SS_E (or SS_{TC}) is reduced by an amount $b_1^2 \sum (X_i - \bar{X})^2$, which is called the sum of squares due to regression and is denoted by SS_R. Equation (1.176) can be written as:

$$SS_E = SS_{TC} - SS_R \tag{1.177}$$

or:

$$SS_T = \sum_i Y_i^2 = n\bar{Y}^2 + SS_E + SS_R = SS_M + SS_E + SS_R \tag{1.178}$$

Thus regression analysis may be looked upon as the process of partitioning the total sum of squares, SS_T into three parts:

(1) The sum of squares due to the mean-SS_M;

(2) The sum of squares due to error SS_E (or deviation about the regression line);

(3) The sum of squares due to regression-SS_R.

Another way of stating this result is that each Y_i value is made up of three parts (or partitioned into three segments), each one leading to the corresponding sum of squares. That is,

$$Y_i = \bar{Y} + \left(\hat{Y}_i - \bar{Y}\right) + \left(Y_i - \hat{Y}_i\right),\ i = 1.2, \ldots, n \tag{1.179}$$

Figure 1.21 shows the partition of Y in graphical form:

It should be noted that the estimated regression line always passes through the point $(\bar{X}, \bar{Y})$. This is obvious from:

$$Y = b_0 + b_1 X_i = \bar{Y} - b_1\bar{X}_i + b_1 X_i \tag{1.180}$$

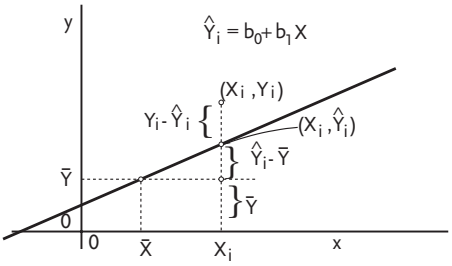

Figure 1.21 Partitioning of total sum of squares in simple linear regression

It can be shown that $SS_E/(n-2)$ is an unbiased estimator of σ^2. Furthermore, SS_E/σ^2 has a χ^2 distribution with n-2 degrees of freedom.

1.6.1.1 Interval Estimation in Simple Linear Regression

Prior to determining confidence intervals or determining test procedures to be used, we recall three assumptions in the model $Y=\beta_0+\beta_1 X$:

- The independent variable X is a fixed variable whose values can be observed without error;
- For any given value of X, Y is normally distributed with mean $\mu_{Y/X}=\beta_0+\beta_1 X$ and variance $\sigma^2_{Y/X} = \sigma^2$.
- That the variance can be represented as $\sigma^2 = \sigma^2_\varepsilon$ which is the same for each X. The estimator for σ^2, as previously mentioned is:

$$S^2 = MS_E = \frac{SS_E}{n-2} = \frac{\sum_i \left(Y_i - \hat{Y}_i\right)^2}{n-2} = \left[\sum_i Y_i^2 - \left(\sum_i Y_i\right)^2 \Big/ n\right] \Big/ n-2 \qquad (1.181)$$

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For a linear regression one can give the interval estimate of parameter β_1 or the slope of the regression line, of its β_0 intercept on the Y-axis, of the true mean Y for any value X ($\mu_{Y/X}=E(Y)$) and the true predicted value, Y_i corresponding to a fixed value of X. The variances of the estimators of these parameters can be shown to be:

$$S^2_{b_1} = \frac{S^2}{\sum_i (X_i - \bar{X})^2} = \frac{S^2}{\sum_i X_i^2 - \left(\sum_i X_i\right)^2 \Big/ n} \qquad (1.182)$$

$$S^2_{b_0} = \left[\frac{1}{n} + \frac{\bar{X}^2}{\sum_i (X_i - \bar{X})^2}\right] \times S^2 = \left(\frac{1}{n} + \frac{(X-\bar{X})^2}{\sum_i X_i^2 - \left(\sum_i X_i\right)^2 \Big/ n}\right) \times S^2 \qquad (1.183)$$

$$S^2_{\widehat{\mu}_{Y/X}} = \left[\frac{1}{n} + \frac{(X-\bar{X})^2}{\sum_i (X_i-\bar{X})^2}\right] \times S^2 = \left[\frac{1}{n} + \frac{(X-\bar{X})^2}{\sum_i X_i^2 - \left(\sum_i X_i\right)^2 \Big/ n}\right] \times S^2 \quad (1.184)$$

$$S^2_{\widehat{Y}} = \left[1 + \frac{1}{n} + \frac{(X-\bar{X})^2}{\sum_i (X_i-\bar{X})^2}\right] \times S^2 = \left[1 + \frac{1}{n} + \frac{(X-\bar{X})^2}{\sum_i X_i^2 - \left(\sum_i X_i\right)^2 \Big/ n}\right] \times S^2 \quad (1.185)$$

For a significance level, α, the (1-α)100% confidence interval for parameter β_1 is:

$$b_1 - S_{b_1} \times t_{n-2;1-\alpha/2} \prec \beta_1 \prec b_1 + S_{b_1} \times t_{n-2;1-\alpha/2} \quad (1.186)$$

For parameter β_0:

$$b_0 - S_{b_0} \times t_{n-2;1-\alpha/2} \prec \beta_0 \prec b_0 + S_{b_0} \times t_{n-2;1-\alpha/2} \quad (1.187)$$

With (1-α) × 100% confidence level, the confidence interval for $\mu_{Y/X}$ is:

$$\widehat{Y} - S_{\widehat{\mu}_{Y/X}} \times t_{n-2;1-\alpha/2} \prec \mu_{Y/X} \prec \widehat{Y} + S_{\widehat{\mu}_{Y/X}} \times t_{n-2;1-\alpha/2} \quad (1.188)$$

The confidence interval for any Y value at the corresponding X value and significance level α is:

$$\widehat{Y} - S_{\widehat{Y}} \times t_{n-2;1-\alpha/2} \prec Y \prec \widehat{Y} + S_{\widehat{Y}} \times t_{n-2;1-\alpha/2} \quad (1.189)$$

It should be noted that the confidence interval for $\mu_{Y/X}$ is narrower than the associated interval for Y, for the latter takes into account the variability of individual Y values. This comes from the fact that:

$$S^2_{\widehat{Y}} = S^2_{\widehat{\mu}_{Y/X}} + S^2 \quad (1.190)$$

or $S^2_{\widehat{Y}} \succ S^2_{\widehat{\mu}_{Y/X}}$. If, after calculating the confidence interval for any parameter, it turns out that it includes the value null, then with a (1-α)100% confidence one can assert that the associated parameter is not essential, i.e. such a parameter is left out of the regression equation.

Example 1.42 [4]

An intermediate step in a reaction process is A→B. While this reaction is carried out at atmospheric pressure, the temperature varies from 1 to 10 °C. As an initial step in the optimization of this process, the relation between conversion of A and temperature must be obtained. Pilot-plant studies have provided the following data:

X:	1	2	3	4	5	6	7	8	9	10
Y:	3	5	7	10	11	14	15	17	20	21

where:
X-temperature, and
Y-is yield

After obtaining the regression model, calculate the confidence intervals for regression parameters.

Assume that the model $Y=\beta_0+\beta_1 X+\varepsilon$ may describe the experimental data in a satisfactory way. This time from experimental data we calculate:

$$\sum X_i = 55.0; \quad \sum Y_i = 123.0; \quad \sum X_i Y_i = 844.0$$

$$\sum X_i^2 = 385.0; \quad \sum Y_i^2 = 1855.0; \quad \left(\sum X_i\right)\left(\sum Y_i\right) = 6765.0$$

$$\left(\sum X_i\right)^2 = 3025.0; \quad \left(\sum Y_i\right)^2 = 15129.0; \quad n = 10$$

$$\bar{X} = \frac{\sum X_i}{n} = 5.5; \quad \bar{Y} = \frac{\sum Y_i}{n} = 12.3$$

$$\sum \left(X_i - \bar{X}\right)\left(Y_i - \bar{Y}\right) = \sum X_i Y_i - \frac{\sum X_i \times \sum Y_i}{n} = 167.5$$

$$\sum \left(X_i - \bar{X}\right)^2 = \sum X_i^2 - \frac{\left(\sum X_i\right)^2}{n} = 82.5$$

$$\sum \left(Y_i - \bar{Y}\right)^2 = \sum Y_i^2 - \frac{\left(\sum Y_i\right)^2}{n} = 342.1$$

$$b_1 = \frac{\sum \left(X_i - \bar{X}\right)\left(Y_i - \bar{Y}\right)}{\sum \left(X_i - \bar{X}\right)^2} = 2.0303; \quad b_0 = \bar{Y} - b_1 \bar{X} = 1.1333$$

$$\widehat{Y} = 1.1333 + 2.0303 \times X$$

$$SS_E = \sum_i \left(Y_i - \bar{Y}\right)^2 - b_1^2 \sum_i \left(X_i - \bar{X}\right)^2 = 2.025$$

$$S^2 = \frac{SS_E}{n-2} = 0.2531; \quad S = \sqrt{S^2} = 0.5031$$

a) 95% confidence interval for β_0 according to Eqs. (1.183) and (1.187):

$$S_{b_0} = \left(\frac{1}{10} + \frac{5.5^2}{82.5}\right)^{1/2} \times 0.031 = 0.3437$$

$$1.1333 - t_{8;0.975} \times S_{b_0} \prec \beta_0 \prec 1.1333 + t_{8;0.975} \times S_{b_0}$$

$1.1333\text{-}2.306 \times 0.3437 < b_0 > 1.1333 + 2.306 \times 0.3437$
$0.3437 < b_0 > 1.9259$

b) 95% confidence interval for β_1 according to Eqs. (1.182) and (1.186):

$$S_{b_1} = \frac{0.5031}{\sqrt{82.5}} = 0.05539$$

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$2.0303\text{-}2.306 \times 0.05539 < \beta_1 < 2.0303 + 2.306 \times 0.05539$
$1.9026 < \beta_1 < 2.1580$

c) 95% confidence interval for $\mu_{Y/X}$ when X=4 according to Eq. (1.184) and (1.188):

$$S_{\widehat{\mu}_{Y/X}} = \left[\frac{1}{10} + \frac{(4-5.5)^2}{82.5}\right]^{1/2} \times 0.5031 = 0.1795$$

$$\widehat{Y} = 1.1333 + 2.0303 \times 4 = 9.2545$$

$9.2545\text{-}2.306 \times 0.1795 < \mu_{Y/X} < 9.2545 + 2.306 \times 0.1795$
$8.8406 < \mu_{Y/X} < 9.6684$

d) 95% confidence interval for Y when X=4 according to Eqs. (1.185) and (1.189):

$$S_{\widehat{Y}} = (1 + 0.1273)^{1/2} \times 0.5031 = 0.5341$$

$9.2545\text{-}2.306 \times 0.5341 < Y < 9.2545 + 2.306 \times 0.5341$
$8.0229 < Y < 10.4861$

Example 1.43 [22]
Monthly consumption of water steam was measured in a production plant. Simultaneously the monthly average temperature of atmosphere was taken. The obtained pairs of values are in the table:

Table 1.67 Water steam consumption

Number of data	1	2	3	4	5	6	7	8	9	10	11	12	13
T [°F]	35.3	29.7	30.8	58.8	61.4	71.3	74.4	76.7	70.7	57.5	46.4	28.9	28.1
Steam consumption	10.98	11.13	12.51	8.40	9.27	8.73	6.36	8.50	7.82	9.14	8.24	12.19	11.88

Number of data	14	15	16	17	18	19	20	21	22	23	24	25
T [°F]	39.1	46.8	48.5	59.3	70.0	70.0	74.5	72.1	58.1	44.6	33.4	28.6
Steam consumption	9.57	10.94	9.58	10.09	8.11	6.83	8.88	7.68	8.47	8.86	10.36	11.08

Determine coefficients of linear regression and 95% confidence intervals for all parameters.

$$\sum X_i = 1315; \quad \sum Y_i = 235.60; \quad \bar{X} = 52.60; \quad \bar{Y} = 9.424;$$

$$\sum X_i Y_i = 11821.4320; \quad \sum X_i^2 = 76323.42; \quad n = 25; \quad \sum Y_i^2 = 2284.1102;$$

$$b_1 = \frac{\sum\sum X_i Y_i - \sum X_i \times \frac{\sum Y_i}{n}}{\sum X_i^2 - \frac{\left(\sum X_i\right)^2}{n}} = \frac{11821.4320 - 1315 \times \frac{235.60}{25}}{76323.42 - \frac{1315^2}{25}} = -0.079829$$

$$b_0 = \bar{Y} - b_1 \times \bar{X} = 9.424 - (-0.079829 \times 52.60)$$

$$\widehat{Y} = 13.623005 - 0.079829 \times X$$

$$\sum \left(Y_i - \bar{Y}\right)^2 = \sum Y_i^2 - \frac{\left(\sum Y_i\right)^2}{n} = 2284.1102 - \frac{235.60^2}{25} = 63.82$$

$$\sum \left(X_i - \bar{X}\right)^2 = \sum X_i^2 - \frac{\left(\sum X_i\right)^2}{n} = 76323.42 - \frac{1315^2}{25} = 7154.42$$

$$SS_E = \sum \left(Y_i - \bar{Y}\right)^2 - b^2 \sum \left(X_i - \bar{X}\right)^2 = 63.82 - (-0.079829)^2 \times 7154.42 = 18.23$$

$$S^2 = \frac{SS_E}{n-2} = \frac{18.23}{23} = 0.7926;\ S = 0.8903$$

a) 95% confidence interval for β_1

$$S_{\widehat{\beta}} = \frac{0.8903}{\sqrt{7154.42}} = 0.0105$$

$-0.079829-t_{23;0.975} \times S_{b1} < \beta_1 < -0.079829+t_{23;0.975} \times S_{b1}$
$-0.079829-2.07 \times 0.0105 < \beta_1 < -0.079829 + 2.07 \times 0.0105$
$-0.1015 < \beta_1 < -0.0581$

b) 95% confidence interval for β_0

$$S_{b_0} = \left(\frac{1}{25} + \frac{52.60}{7154.42}\right)^{1/2} \times 0.8903 = 0.3799$$

$13.623005-2.07 \times 0.3799 < \beta_1 < 13.623005 + 2.07 \times 0.3799$
$12.836612 < \beta_1 < 14.409398$

c) 95% confidence interval for the response mean $\mu_{Y/X}$ when $X = \bar{X}$:

$$S_{\widehat{\mu}_{Y/X=\bar{X}}} = \left(\frac{1}{25} + 0.0\right)^{1/2} \times 0.8903 = 0.1781 \quad ;$$

$$\widehat{Y} = \bar{Y} = 9.424;\quad \bar{X} = 52.60;$$

$$9.424 - 2.07 \times 0.1781 \prec \widehat{\mu}_{Y/X=\bar{X}} \prec 9.424 + 2.07 \times 0.1781 \quad ;$$
$$9.05531 \prec \widehat{\mu}_{Y/X=\bar{X}} \prec 9.7927$$

The standard deviation for estimating the mean Y has a minimal value for $X = \bar{X}$, and it rises when X takes values on either side of $\bar{X}$. In other words, the farther away X was taken from the mean $\bar{X}$, the greater the error in estimating the mean Y for a given X value may be expected. It is obvious if we take the following X value:

X=28.60; $S_{\widehat{\mu}_{Y/X=\bar{X}}}$ =0.3091 ; $8.7842 \prec \widehat{\mu}_{Y/X} \prec 10.0638$

Provided we continue this calculation we shall obtain its geometric interpretation as shown in Figure 1.22.

Both the figure and previous calculation show that the width of 95% confidence interval round the regression line changes depending on the value X takes. The obtained limit lines of the 95% confidence interval are hyperbolas.

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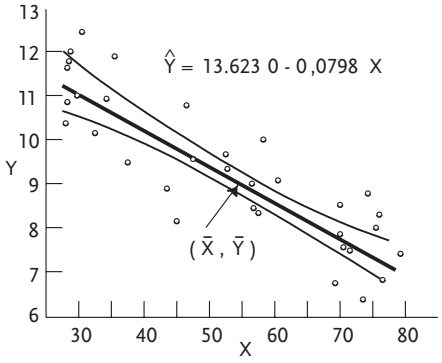

Figure 1.22 Geometric presentation of 95% confidence interval

1.6.1.2 **Analysis of Variance of Simple Linear Regression**

We have previously introduced the sum of squares due to error as $MS_E=SS_E/(n-2)$ and said that it is the unbiased estimate of error variance σ^2 because $E(MS_E)=\sigma^2$ no matter whether the null hypothesis $H_0:\beta_1=0$ is correct or not. It is easy to prove that the expected value of the regression mean square, $MS_R=SS_R/1$, is the biased estimate of variance σ^2 if not $\beta_1=0$. This can be written in the form:

$$E(MS_R) = E\left(\frac{SS_R}{1}\right) = \sigma^2 + \beta_1^2 \sum \left(X_i - \bar{X}\right)^2 \succ \sigma^2 \tag{1.191}$$

The two above-mentioned expected values of the mean squares suggest introduction of F-test for testing the null hypothesis H_0: $\beta_1=0$.

$$F = \frac{MS_R}{MS_E} \tag{1.192}$$

The given ratio of mean squares due to regression and error has F-distribution with $f_1=1$ and $f_2=n-2$ degrees of freedom if the null hypothesis $H_0:\beta_1=0$ is correct. The null hypothesis is rejected if the obtained value $F>F_{1;n-2;1-\alpha}$ is above the tabular one. Since MS_R and MS_E are the estimates σ^2 provided $H_0:\beta_1=0$, then the null hypothesis with α significance level is rejected if the F-ratio is considerably above 1 for $E(MS_R)>\sigma^2$ when $H_0:\beta_1=0$ is not true.

Analysis of variance for the linear regression is given in the usual way in Table 1.68.

Apart from the mentioned one, the analysis of variance is also frequently used where the total sum of squares SS_T is corrected for the mean sum of squares SS_M. Such a sum of squares is called the total corrected sum of squares $SS_{TC}=SS_T-SS_M$. Analysis of variance of the linear regression with total corrected sum of squares is given in Table 1.69.

Table 1.68 AOV of linear regression

Source of variation	f	SS	MS	E(MS)	Test statistic
Average ($_0$)	1	$n\bar{Y}^2 = SS_M$	$MS_M = SS_M$	–	–
Regression (β_1)	1	$b_1 \sum_i (X_i - \bar{X})(Y_i - \bar{Y}) = SS_R$	$MS_R = SS_R$	$\sigma^2 + \beta_1^2 \sum_i (X_i - \bar{X})^2$	$\frac{MS_R}{MS_E}$
Error or residual	n-2	$\sum_i \left(Y_i - \widehat{Y}_i\right)^2 = SS_E$	$MS_E = SS_E/(n-2)$	σ^2	
Total	n	$\sum_i Y_i^2 = SS_T$	–	–	–

Table 1.69 AOV of linear regression

	f	SS	MS	Test statistic
Regression (β_1)	1	$b_1 \left[\sum_i X_i Y_i - \left(\sum_i X_i\right)\left(\sum_i Y_i\right) \Big/ n\right]$	$MS_R = SS_R$	$\frac{MS_R}{MS_E}$
Error or residual	n-2	$\sum_i \left(Y_i - \widehat{Y}_i\right)^2$	$MS_E = \frac{SS_E}{(n-2)}$	–
Total corrected sum	n-1	$\sum_i Y_i^2 - \left(\sum_i Y_i\right)^2 \Big/ n$	–	–

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Different from the previous one, this table of analysis of variance introduces different expressions for sums of squares that are more suitable for calculations.

Example 1.44

Do analysis of variance for the regression analysis in Example 1.42, or the obtained linear regression.

The calculated value of analysis of variance is F=1343.6 for the null hypothesis H_0:β_1=0. However, since the tabular value is $F_{1;8;0.95}$=5.32 the null hypothesis is rejected and the alternative hypothesis accepted that the regression coefficient β_1 with 95% confidence level is statistically significant.

Table 1.70 Analysis of variance

Source of variation	f	SS	MS	E(MS)	Test statistic
Average β_0	1	1512.900	1512.900	–	–
Regression β_1	1	340.075	340.075	$\sigma^2 + 82,5 \cdot \hat{\beta}_1^2$	$\frac{340{,}075}{0{,}2531} = 1343,6$
Residual	8	2.025	0.2531	σ^2	–
Total	10	1855.000	–	–	–

Example 1.45

Do analysis of variance for the linear regression obtained from experimental value in Example 1.43.

Table 1.71 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Regression β_1	1	45.59	45.59	57.52	$F_{1;23;0.95}$=4.28
Residual	23	18.23	0.7926	–	–
Corrected total	24	63.82	–	–	–

Since the calculated value is F=57.52>F_{TAB}=4.28 it can be with 95% confidence level asserted that the regression coefficient β_1 is statistically greater than zero and that it should be kept in the linear regression.

1.6.1.3 Lack of Fit of the Simple Linear Regression[4)]

The test for lack of fit of the regression model breaks up the residual sum of squares into a sum of squares for lack of fit and an experimental error sum of squares. This can be done only if we have some values of X for which we have more than one value for Y. This concept of separating a sum of squares or experimental error variance from the residual sum of squares or residual variance has been known from the chapter on analysis of variance. It should be noted that replicating the trial in this case, and in general, does not mean keeping the X value in the experiment constant and reading Y response several times, but a literal taking of the same X value in different times and measuring or single reading of Y each time. Thereby experiments or trials are done between such times for other X values. Suppose that of the n Xs there are k distinct Xs, where k<n, which occur with frequencies n_1,n_2, ...,n_k , where $n_1+n_2+...+n_k=n$. The sum of squares of the n_i, Ys corresponding to an X_i:

$$\sum_{j=1}^{n_i} \left(Y_j - \bar{Y}\right)^2 = \sum_{j=1}^{n_i} Y_j^2 - n_i \bar{Y}^2 = (SS_E)_i \tag{1.193}$$

4) Adequacy of linear regression

is a sum of squares due to experimental error. The pooled sum of squares due to experimental error is denoted by:

$$SS_E = \sum_{i=1}^{k} (SS_E)_i \tag{1.194}$$

with next degrees of freedom:

$$\sum_{i=1}^{k} (n_i - 1) = n - k \tag{1.195}$$

The mean square due to experimental error is:

$$MS_E = \frac{SS_E}{n-k} \tag{1.196}$$

The usual residual sum of squares is:

$$SS_{RES.} = \sum_{i=1}^{n} \left(Y_i - \widehat{Y}\right)^2$$

with (n-2) degrees of freedom. The lack of fit of sum of squares is:

$$SS_{LF}=SS_{RES.}-SS_E \tag{1.197}$$

with n-2-(n-k)=k-2 degrees of freedom or the mean lack of fit of sum of squares is:

$$MS_{LF} = \frac{SS_{LF}}{k-2} \tag{1.198}$$

The critical region in testing H_0: Lack of fit is $F \rangle F_{k-2,n-k,1-\alpha}$ where:

$$F = \frac{MS_{LF}}{MS_E} \tag{1.199}$$

has an F-distribution with k-2 and n-k degrees of freedom.

If the obtained value of F-ratio is below the tabular value $F_{k\text{-}2;n\text{-}k;1\text{-}\alpha}$ then the null hypothesis that the linear regression is adequate to (1-α) 100% confidence level is accepted. Hence the linear regression variance analysis should also include check of lack of fit of linear regression. If in variance analysis of F-ratio for lack of fit is statistically:

- Significant, it shows that the obtained linear regression does not adequately describe the experimental results. The next step is establishing the reason for lack of fit.
- Insignificant, there is no reason to doubt lack of fit of the obtained regression model, and mean sums of squares of experimental error and lack of fit can be used for variance estimate σ^2.

Some of the standard situations in doing regression analysis are given in Fig. 1.23.

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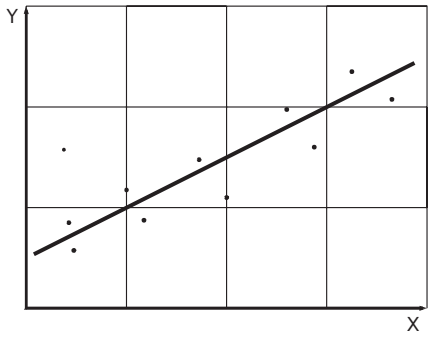
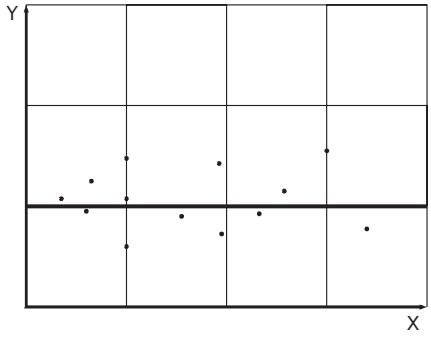


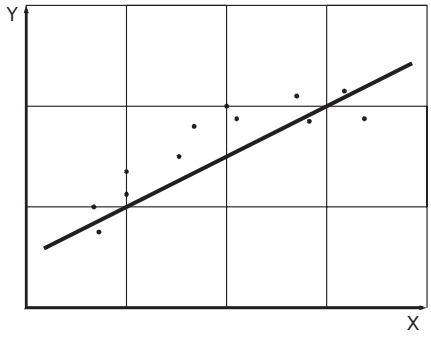
Example 1

- Model : $Y = \beta_0 + \beta_1 X + \varepsilon$
- No lack of fit
- Significant linear regression
- Use model: $\widehat{Y} = b_0 + b_1 X$



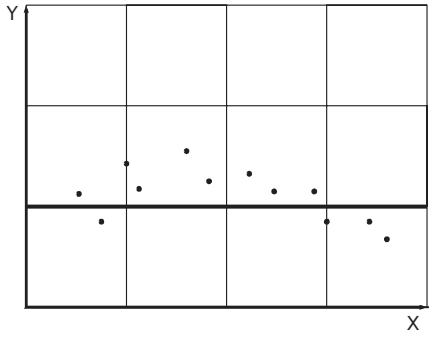
Example 2

- Model: $Y = \beta_0 + \beta_1 X + \varepsilon$
- No lack of fit
- Linear regression not significant
- Use model: $\widehat{Y} = \bar{Y}$



Example 3

- Model: $Y = \beta_0 + \beta_1 X + \varepsilon$
- Significant lack of fit
- Significant linear regression
- Use model: $\widehat{Y} = b_0 + b_1 X + b_{11} X^2$



Example 4

- Model: $Y = \beta_0 + \beta_1 X + \varepsilon$
- Significant lack of fit
- Linear regression not significant
- Use model: $\widehat{Y} = b_0 + b_1 X + b_{11} X^2$

Figure 1.23 Typical linear regression situations

Example 1.46 [22]

To demonstrate analysis of variance for linear regression model, the following experimental results were used:

Table 1.72 Experimental results

No	X	Y	No	X	Y	No	X	Y
1	1.3	2.3	**9**	3.7	1.7	**17**	5.3	3.5
2	1.3	1.8	**10**	4.0	2.8	**18**	5.3	2.8
3	2.0	2.8	**11**	4.0	2.8	**19**	5.3	2.1
4	2.0	1.5	**12**	4.0	2.2	**20**	5.7	3.4
5	2.7	2.2	**13**	4.7	5.4	**21**	6.0	3.2
6	3.3	3.8	**14**	4.7	3.2	**22**	6.0	3.0
7	3.3	1.8	**15**	4.7	1.9	**23**	6.3	3.0
8	3.7	3.7	**16**	5.0	1.8	**24**	6.7	5.9

After calculating the given experimental data the following linear regression is obtained: $\widehat{Y} = 1.436 + 0.338X$. Do the analysis of variance by determing the lack of fit of the regression model.

Analysis of variance with no setting apart of the residual variance into experimental error and lack of fit variance is given in the table:

Table 1.73 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Regression β_1	1	6.326	6.326	6.569	$F_{1;22;0.95}$=4.30
Residual	22	21.192	0.963	–	–
Corrected total	23	27.518	–	–	–

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The procedure of calculating the experimental error variance or the so-called pure error consists of:

S_E for X=1.3 is:

$$(SS_E)_{X=1.3} = \sum_{j=1}^{n_{X=1.3}} Y_j^2 - n_{X=1.3} \times \bar{Y}^2 = 2.3^2 + 1.8^2 - 2\left(\frac{2.3+1.8}{2}\right)^2$$
$$= 0.5(2.3 - 1.8)^2 = 0.125$$

with degrees of freedom f=n-k=2-1=1;

SS_E for X=4.7 is:

$$(SS_E)_{X=4.7} = \sum_{j=1}^{n_{X=4,}} Y_j^2 - n_{X=4.7} \times \bar{Y}^2 = 5.4^2 + 3.2^2 + 1.9^2 - 3\left(\frac{5.4+3.2+1.9}{3}\right)^2$$
$$= 6.26$$

with degree of freedom f=n-k=3-1=2

After continuing such calculations for other replicated trials we obtain:

Table 1.74 Analysis of variance

Value X	$\sum_{j}^{n_i}\left(Y_j-\bar{Y}\right)^2$	Degrees of freedom f
1.3	0.125	1
2.0	0.845	1
3.3	2.000	1
3.7	2.000	1
4.0	0.240	2
4.7	6.260	2
5.3	0.980	2
6.0	0.020	1
Total	12.470	11

Now that the sum of squares of experimental error SS_E =12.470 with the degree of freedom f=11; has been calculated, we can offer the analysis of variance table with the lack of fit variance:

Table 1.75 Analysis of variance

Source of variance	f	SS	MS	F	F_T
Regression β_1	1	6.326	6 ,326	$\frac{6.326}{0.963}=6.569$	$F_{1;22;0.95}$=4.30
Residual	22	21.192	0.963	–	–
Corrected total	23	27.518	–	–	–
Lack of fit	11	8.722	0.793	$\frac{0.793}{1.134}=0.699$	$F_{11;11;0.95}$=2.82
Pure error	11	12.470	1.134	–	–

Due to the fact that the calculated value of F=0.699<$F_{11;11;0.95}$=2.82 there is no reason to doubt the lack of fit of the linear regression.

1.6.2 Multiple Regression

Multiple regression can be used to develop a quantitative equation relating a dependent variable with several independent variables. In multiple linear regression, any number of independent variables may be considered:

$$Y=\beta_0+\beta_1X_1+\beta_2X_2+\ldots+\beta_pX_p+\varepsilon \tag{1.200}$$

Assumptions of a multiple regression analysis are identical to those for linear regression except for the p independent variables in this case. To reach regression coefficient estimates b_i by the method of least squares, we again have to minimize

the sum of squares due to errors. Similarly, as in linear regression, we have n data for Y and X_1 ,X_2 ,...X_p , therefore sum of squares of errors:

$$SS_E = \sum_i e_i^2 = \sum_i \left(Y_i - \widehat{Y}_i\right)^2 = \sum_i \left(Y_i - b_0 - b_1 X_{1i} - b_2 X_{2i} - \ldots - b_p X_{pi}\right) \quad (1.201)$$

The sum of squared errors between the observed value and predicted value is minimized by taking the partial derivative $(SS_E)/b_i$ with respect to each parameter and setting each result equal to zero:

$$nb_0 + b_1 \sum X_{1i} + b_2 \sum X_{2i} + \ldots + b_p \sum X_{pi} = \sum Y_i$$

$$b_0 \sum X_{1i} + b_1 \sum X_{1i}^2 + b_2 \sum X_{1i} X_{2i} + \ldots + b_p \sum X_{1i} X_{pi} = \sum X_{1i} Y_i$$

$$b_0 \sum X_{2i} + b_1 \sum X_{1i} X_{2i} + b_2 \sum X_{2i}^2 + \ldots + b_p \sum X_{2i} X_{pi} = \sum X_{2i} Y_i$$

$$b_0 \dot{\sum} X_{pi} + b_1 \dot{\sum} X_{1i} X_{pi} + b_2 \dot{\sum} X_{2i} X_{pi} + \ldots + b_p \dot{\sum} X_{pi}^2 = \dot{\sum} X_{pi} Y_i$$

here the values are summed for all i=1,..., n data points. To obtain regression coefficient estimates b_0, b_1, ... , b_p it is necessary to solve the given simultaneous system of linear equations. The simplest way for this is to use matrix algebra or digital computers. In the case of p independent variables the sum of squares due to error is:

$$SS_E = \sum_i \left(Y_i - \widehat{Y}_i\right)^2$$

where:

$$\widehat{Y}_i = b_0 + b_1 X_1 + \ldots + b_p X_p$$

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It is easy to prove:

$$SS_E = \sum_i \left(Y_i - \widehat{Y}_i\right)^2 = \sum_i \left(Y_i - \bar{Y}_i\right)^2 - \left[b_1 \sum_i \left(X_{1i} - \bar{X}_1\right)\left(Y_i - \bar{Y}_i\right) + \ldots + b_{pi} \sum_i \left(X_{pi} - \bar{X}_p\right)\left(Y_i - \bar{Y}_i\right)\right] \quad (1.202)$$

where:

$$\bar{X}_j = \sum_{i=1}^{n} \frac{X_{ji}}{n}.$$

The sum that is in the central parenthesis is the sum of squares due to regression SS_R so that:

$SS_E = SS_{TC} - SS_R$; $SS_{TC} = SS_E + SS_R$

where:

$$SS_R = b_1 \sum \left(X_{1i} - \bar{X}_1\right)\left(Y_i - \bar{Y}\right) + \ldots + b_p \sum \left(X_{pi} - \bar{X}_p\right)\left(Y_i - \bar{Y}\right) \quad (1.203)$$

It is of particular importance to do statistical testing of the null hypothesis:

H_0: $b_1=b_2=...=b_p=0$

Testing this null hypothesis is done by analysis of variance of multiple regression shown in Table 1.76. It should also be noted that the unbiased estimate σ^2 is given as:

$$MS_E = \frac{\sum \left(Y_i - \widehat{Y}_i\right)^2}{n-p-1}.$$

Table 1.76 Analysis of varaince of multiple regression

Source of variation	f	SS	MS	Test statistic
Regression	p	SS_R	$MS_R=SS_R/p$	MS_R/MS_E
Error-residual	n-p-1	SS_E	$MS_E=SS_E/(n-p-1)$	–
Total	n-1	SS_{TC}	–	–

The test statistic is the $F=MS_R/MS_E$ ratio which is compared to the tabular value $F_{p;(n-p-1);1-\alpha}$. If: $F>F_{p;(n-p-1);1-\alpha}$ the null hypothesis is rejected and the alternative one accepted.

Example 1.47 [4]

It is necessary to relate the per cent gas absorbed in a tower, Y, to the gas temperature X_1 and the vapor pressure of the absorbing liquid, X_2 . The following data are available:

Table 1.77 Experimental values

Y	X_1	X_2
6.0	113.5	3.2
10.0	130.0	4.8
20.0	154.0	8.4
30.0	169.0	12.0
50.0	187.0	18.5
80.0	206.0	27.5
100.0	214.0	32.0

The postulated model is $Y=\beta_0+\beta_1X_1+\beta_2X_2+\varepsilon$. Determine regression coefficients and do analysis of variance of multiple regression. From tabulated data the following values are calculated:

$$\sum X_{1i} = 1251.5; \quad \sum X_{2i} = 107.9; \quad \sum X_{1i}X_{2i} = 20359.0;$$

$$\sum X_{1i}^2 = 211344.25; \quad \sum X_{2i}^2 = 2371.339; \quad \sum X_{1i} \sum X_{2i} = 134411.0;$$

$$\left(\sum X_{1i}\right)^2 = 1566252.25; \quad \left(\sum X_{2i}\right)^2 = 11534.75; \quad \sum Y_i = 297.5;$$

$$\sum X_{1i} Y_i = 57478.0; \quad \sum X_{2i} Y_i = 6971.70; \quad \sum Y_i^2 = 20338.25;$$

$$\sum X_{1i} \sum Y_i = 372321.25; \quad \sum X_{2i} \sum Y_i = 31951.49;$$

$$\bar{X}_1 = 156.4375; \quad \bar{X}_2 = 13.4249; \quad \bar{Y} = 37.1875$$

$$\sum \left(X_{1i} - \bar{X}_1\right)^2 = 15562.75; \quad \sum \left(X_{2i} - \bar{X}_2\right)^2 = 929.4951;$$

$$\sum \left(X_{1i} - \bar{X}_1\right)\left(X_{2i} - \bar{X}_2\right) = 3557.914$$

$$\sum \left(X_{1i} - \bar{X}_1\right)\left(Y_i - \bar{Y}\right) = 10937.84; \quad \sum \left(X_{2i} - \bar{X}_2\right)\left(Y_i - \bar{Y}\right) = 2927.762;$$

$$\sum \left(Y_i - \bar{Y}_i\right)^2 = 9274.968$$

Regression coefficients are obtained by solving the system of equations:

$$\begin{cases} 8b_0 + 1251.5b_1 + 107.9b_2 = 297.5 \\ 1251.5b_0 + 211344.25b_1 + 134411.00b_2 = 57478.0 \\ 107.9b_0 + 134411.00b_1 + 11534.75b_2 = 31951.49 \end{cases}.$$

b_1=-0.13840 ; b_2=3.6796 ; b_0=9.4398 ;

Linear regression has this form:

$$\widehat{Y} = 9.4398 - 0.1384 \times X_1 + 3.6796 \times X_2$$

To analyze the variance we calculate the sum of squares due to regression.

$$SS_R = b_1 \sum \left(X_{1i} - \bar{X}_1\right)\left(Y_i - \bar{Y}\right) + b_2 \sum \left(X_{2i} - \bar{X}_2\right)\left(Y_i - \bar{Y}\right)$$

$$= -0.1384 \times 10937.84 + 3.6796 \times 2927.762 = 9259.196$$

and the sum of squares due to error:

$$SS_E = \sum \left(Y_i - \widehat{Y}\right)^2 = \sum \left(Y_i - \bar{Y}\right)^2 - SS_R = 9274.968 - 9259.196 = 15.772$$

Table 1.78 Analysis of variance

Source of variation	f	SS	MS	F	F_T
Regression	2	9259.196	4629.598	1467.850	$F_{2;5;0.95}$=5.79
Residual-error	5	15.772	3.154	–	–
Total	7	9274.968	–	–	–

Based on analysis of variance with 95% confidence level the null hypothesis: H_0: $\beta_1=\beta_2=0$ is rejected, for the calculated value F =4629.598/3.154 = 1467.850 is considerably above the tabular value $F_{2;5;0.95}$=5.79.

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1.6.3 Polynomial Regression

In the case of polynomial or curvilinear regression, as given by the model:

$$Y=\beta_0+\beta_1X+\beta_2X^2+\beta_pX^p+...+\varepsilon \tag{1.204}$$

there is only one independent variable: X. However, p-1 other independent variables are defined as powers of X. This kind of p-degree polynomial regression may be brought down to multiple linear regression by introducing these changes: $W_1=X$; $W_2=X^2$;... ;$W_p=X^p$. In a multiple regression with independent variables W_p, we determine the regression coefficients by the procedure given in the previous section. The polynomial regression is reduced to the multiple linear regression model given by Eq. (1.204) with independent variables W_1, W_2, ...,W_p.

As an example consider the quadratic model:

$$Y=\beta_0+\beta_1X+\beta_2X^2+\varepsilon \tag{1.205}$$

In this case $W_1=X$ and $W_2=X^2$ and the normal equations are, given n observations on X and Y:

$$\begin{cases} nb_0 + b_1 \sum_i W_{1i} + b_2 \sum_i W_{2i} = \sum_i Y_i \\ b_0 \sum_i W_{1i} + b_1 \sum_i W_{1i}^2 + b_2 \sum_i W_{1i} W_{2i} = \sum_i W_{1i} Y_i \\ b_0 \sum_i W_{2i} + b_1 \sum_i W_{1i} W_{2i} + b_2 \sum_i W_{2i}^2 = \sum_i W_{2i} Y_i \end{cases} \tag{1.206}$$

However,

$$W_{1i} = X_i;\ W_{2i} = X_i^2$$

Thus the normal equations become:

$$\begin{cases} nb_0 + b_1 \sum_i X_i + b_2 \sum_i X_i^2 = \sum_i Y_i \\ b_0 \sum_i X_i + b_1 \sum_i X_i^2 + b_2 \sum_i X_i^3 = \sum_i X_i Y_i \\ b_0 \sum X_i^2 + b_1 \sum X_i^3 + b_2 \sum X_i^4 = \sum X_i^2 Y_i \end{cases} \tag{1.207}$$

The equations (1.207) can be solved for b_0, b_1 and b_2. Extensions to polynomials of higher degree are obvious and the solution follows in the same manner.

It should be pointed out that when one speaks of a linear model in regression the term linear means linear in the parameters β_0, β_1,..., β_p and not in the independent variable X. Other examples of linear models (linear in the parameters) are:

$$Y = \beta_0 + \beta_1 \log X + \beta_2 X^2 + \varepsilon$$
$$Y = \beta_0 + \beta_1 e^{-X} + \beta_2 X^{1/2} + \varepsilon$$
$$Y = \beta_0 + \beta_1 e^{-X_1} + \beta_2 X_2^2 + \beta_3 X_3 + \varepsilon$$

The mentioned regressions are linear by their regression coefficients but not by their independent variables. Nonlinearity by independent variables is easily brought down to linearity through these changes:

$$W_1 = \log X; \quad W_2 = X^2; \quad \Rightarrow \quad Y = \beta_0 + \beta_1 W_1 + \beta_2 W_2 + \varepsilon$$

$$W_1 = e^{-X}; \quad W_2 = X^{1/2}; \quad \Rightarrow \quad Y = \beta_0 + \beta_1 W_1 + \beta_2 W_2 + \varepsilon$$

$$W_1 = e^{-X_1}; \quad W_2 = X_2^2; \quad W_3 = X_3; \quad \Rightarrow \quad Y = \beta_0 + \beta_1 W_1 + \beta_2 W_2 + \beta_3 W_3 + \varepsilon$$

All regression equations, linear by their regression coefficients, are analyzed by thus far developed methods. If an equation is not linear by coefficients we then deal with nonlinear regression equations, the analysis of which is very complicated and requires iterative procedures.

For instance, *Van Laar's* equation for the coefficient of activity of binomial mixture or the steam-liquid balance, is nonlinear by coefficients A and B, as shown in the equation.

$$\log\gamma_1 = \frac{A}{\sqrt{1+\frac{AX_1}{(1-X_1)B}}} \tag{1.208}$$

Suppose we have a set of empirical-experimental data for two variables and that the obtained data may be described by linear regressions. Which of the linear regressions can describe the experimental data will be known after checking their lack of fit. It should be pointed out that a large number of linear regressions are at our disposal and that some of them have for this particular case been given in Fig. 1.24.

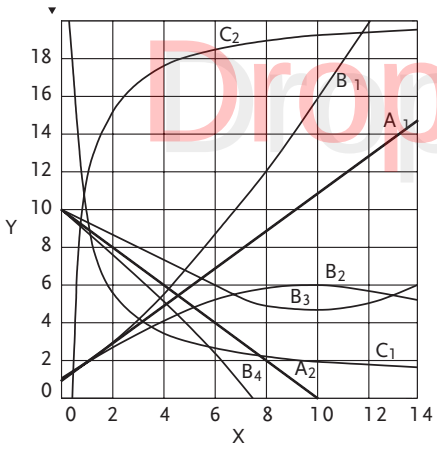

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Figure 1.24 Useful forms for empirical equations

Tabular values of linear regression coefficients for regressions shown in Fig. 1.24 are given in Table 1.79.

Table 1.79 Regression coefficients of linear regressions

Regression	General form	Regression coefficients			Curve
		b_0	b_1	b_2	
A	$Y=b_0+b_1X$	1	1	·	A_1
		10	-1	·	A_2
		1	1	0.05	B_1
B	$Y=b_0+b_1X+b_2X^2$	1	1	-0.05	B_2
		10	-1	0.05	B3
		10	-1	-0.05	B_4
C	$Y=b_0+b_1/X$	1	10	·	C_1
		20	-10	·	C_2

One can notice in Fig. 1.24 that the form of some linear regressions, especially group B, is very sensitive to a change in regression coefficient value.

Example 1.48 [4]
It is believed that the effect of temperature on catalyst activity is quadratic. The proposed model is:

$Y=\beta_0+\beta_1X+\beta_2X^2+\varepsilon$.

Eight different temperatures (coded X data below) were used. The resulting activities are given as Y. Determine the polynomial regression coefficients.

Y:	0.846;	0.573;	0.401;	0.288;	0.209;	0.153;	0.111;	0.078;
X:	2;	4;	6;	8;	10;	12;	14;	16;

If we let $W_1=X$ and $W_2=X^2$, the model reduces to the form

$Y=\beta_0+ \beta_1W +\beta_2W_2+\varepsilon$

Following the same procedure as for multiple linear regression, the values of the regression coefficients are:

$b_0=1.05652$; $b_1=-0.13114$; $b_2=0.00447$

The resulting regression equation is then:

$$\widehat{Y} = 1.05652 - 0.13114 \times X + 0.00447 \times X^2$$

Example 1.49 [23]
We will develop an equation from the data on the heat capacity of benzene vapor as a function of temperature. Experimental values are shown in an accompanying table:

C_p cal/K gmol:	19.65;	26.74;	32.80;	37.74;	41.75;	45.06;	47.83;	50.16;
T K	300;	400;	500;	600;	700;	800;	900;	1000.

Based on analysis of point distribution on a *scatter diagram*, the plotted data are clearly not linear, so a three-term polynomial will be used:

$C_p=b_0+b_1T+b_2T^2$
$C_p=b_0+b_1T+b_21/T$
$C_p=b_0+b_1T$
$C_p=b_1T$

Calculate the linear regression coefficients for all given regression models and present them graphically.. The linear regression $C_p=\beta_0+\beta_1T+\beta_2T^2$ is brought by the method of least squares down to the following system of normal equations:

$$\begin{cases} nb_0 + b_1 \sum T + b_2 \sum T^2 = \sum C_p \\ b_0 \sum T + b_1 \sum T^2 + b_2 \sum T^3 = \sum C_p T \\ b_0 \sum T^2 + b_1 \sum T^3 + b_2 \sum T^4 = \sum C_p T^2 \end{cases}$$

From the data given: n=8

$\sum C_p$=19.65+...+50.16=301.73

$\sum T$=300+...+1000=5200

$\sum T^2=300^2+...+1000^2=3.8\times10^6$

$\sum T^3=300^3+...+1000^3=3.016\times10^9$

$\sum T^4=300^4+...+1000^4=2.5316\times10^{12}$

$\sum C_p T$=19.65×300+...+50.16×1000=214115

$\sum C_p T^2=19.65\times300^2+...+50.16\times1000^2=166.0315\times10^6$

Substituting these values in normal equations we get the regression coefficients:

b_0=-13.212 ; b_1=0.12395 ; $b_2=-6.24\times10^{-5}$

so that the regression becomes:

$C_p=-13.212+0.12395T-6.2400\times10^{-5}T^2$

Calculations for all plotted regressions are in the next table.

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Table 1.80 Calculation and experimental values

Temperature [K]	Heat capacity [cal/gmol K]	Calculated data for regressions			
		A	B	C	D
300	19.65	18.36	18.08	22.72	16.90
400	26.74	26.38	27.53	27.01	22.54
500	32.80	33.16	33.66	31.29	28.17
600	37.74	38.70	38.25	35.57	33.81
700	41.75	42.98	41.80	39.86	39.44
800	45.06	46.01	44.82	44.14	45.08
900	47.83	47.80	47.44	48.42	50.71
1000	50.16	48.34	49.89	52.71	56.35

A geometric interpretation of all four linear regressions is given in Fig. 1.25.

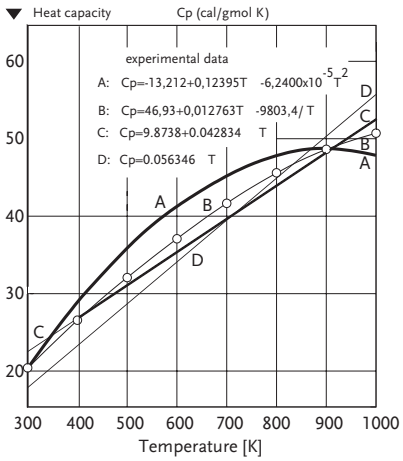

Figure 1.25 Compare the fit between the data and the plotted regression equations

1.6.4
Nonlinear Regression

We often meet mathematical models in engineering practice that are not linear either by their regression coefficients or their independent variables. The non linearity by their independent variables belongs to polynomial analysis, and this was elaborated in the previous section. Nonlinearity by regression coefficients, however, is a heavier problem and it is nowadays solved by iterative procedure helped by fast digital computers. The procedures of determining regression coefficients in nonlinear regressions itself is given in reference [22].

A nonlinear model that occurs quite frequently is:

$$Y = \beta_0 \times e^{\beta_1 X} \tag{1.209}$$

This model is usually handled by means of taking the natural log of both sides of the equations yielding.

$$\ln Y = \ln\beta_0 + \beta_1 X \tag{1.210}$$

Letting $Z=\ln Y$, $\alpha_0=\ln\beta_0$ and $\beta_1=\beta_1$, the model thus reduces to the linear model:

$$Z = \alpha_0 + \alpha_1 X \tag{1.211}$$

Nowadays the method of least squares is applied to determine the regression coefficients α_0 and α_1.

The following nonlinear method is also met in practice:

$$Y = \beta_0 \beta_1^{X} \tag{1.212}$$

This nonlinear model becomes linear when logarithms and substitutions are introduced.

$$\log Y = \log\beta_0 + X \log\beta_1 \tag{1.213}$$

Substitutions:

$$Z = \log Y, \qquad \log\beta_0 = \alpha_0; \qquad \log\beta_1 = \alpha_1 \qquad Z = \alpha_0 + \alpha_1 X$$

One should be careful in using transformations such as the above, since if it is assumed that the original variable is normally distributed, then the transformed variable may not be. The homogeneity of variance property may be likewise violated. Frequently, however, the original assumption of normality may not be justified and the transformed variables have a distribution closer to normal.

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Example 1.50 [4]

The vapor pressure of water absorbed on silica gel can be expressed as a function of the vapor pressure of pure water for various gel loadings in spacecraft humidity-water-recovering systems. For the water loading of 0.1 lb water/lb dry silica gel, the following data were obtained:

p, absorbed H_20:	0.038;	0.080;	0.174;	0.448;	1.43;	5.13;	9.47;
p, pure H_2O:	0.2	0.4	0.8	2.0	6.0	20.0	35.0

A plot of the p data on *Log-Log* paper yields a straight line so an equation of the form:

$$Y = \beta_0 X^{\beta_1}.$$

By applying logarithms and the following substitutions we get the linear regression model:

$$\log Y = \log\beta_0 + \beta_1 \log X$$

Substitutions: $Z=\log Y$, $\alpha_0=\log\beta_0$, $W=\log X$ so that:

$$Z = \alpha_0 + \beta_1 W$$

The following quantities are then calculated:

$$\bar{Z} = -0.254785; \quad \bar{W} = 0.390065; \quad \sum Z = -1.783492; \quad \sum W = 2.730458$$

$$\sum Z^2 = 5.400247; \quad \sum W^2 = 5.429265; \quad \sum ZW = 3.950120; \quad \left(\sum W\right)^2 = 7.455401;$$

$$\left(\sum Z\right)^2 = 3.3180843; \quad \sum Z \sum W = -4.869750.$$

Coefficients are obtained by solving normal equations.

Z=-0.670018+1.06452W

By conversion into original variables we get:

$\widehat{Y} = 0.21379 \times X^{1.06452}$

Example 1.51

In a pilot plant for producing composite rocket propellants a batch of propellant was produced with the idea of characterizing it by measuring the linear burning rate at different pressures in *Crawford's* bomb. The following values were obtained experimentally:

V mm/s:	11.71;	13.63;	17.25;	18.92;
P bar :	50.6;	75.3;	116.3;	138.8.

Based on theoretical and empirical knowledge the relationship between burning rate and pressure had the form:

$V=bP^n$

Determine the coefficients in the given nonlinear regression model.
The obtained values were:

b=1.744
n=0.482.

1.7 Correlation Analysis

Having determined that a relationship exists between variables, the next question that arises is that of how closely the variables are associated. The strongest and closest relationship between variables is the functional relationship, i.e. the relationship where each value of one independent variable corresponds to the exact value of a dependent variable. A weak relationship between variables, subject to smaller or greater diversions, is called *correlation or stochastic*.

The statistical techniques that have been developed to measure the amount of association between variables are called *correlation methods*. A statistical analysis performed to determine the degree of correlation is called a *correlation analysis*. For

example, the circle area and its radius are functionally connected, while the variables that give the burning rate and pressure of a propellant show a correlational and stochastic relationship. The term used to measure correlation is referred to as a *correlation coefficient*. The correlation coefficient measures how well the regression equation fits the experimental data. As such, it is closely related to the standard error of estimate.

It has been mentioned before that the first orientation on the form of relationship between variables is given by graphic presentation of experimental values in a coordinate system. Such a graph is called a *scatter diagram*. The distribution of points in the scatter diagram determines the direction and form of the relationship and up to a point its strength. Fig. 1.26 shows cases of *positively stronger* and *negatively weaker* linear correlations as well as cases of *noncorrelation*;

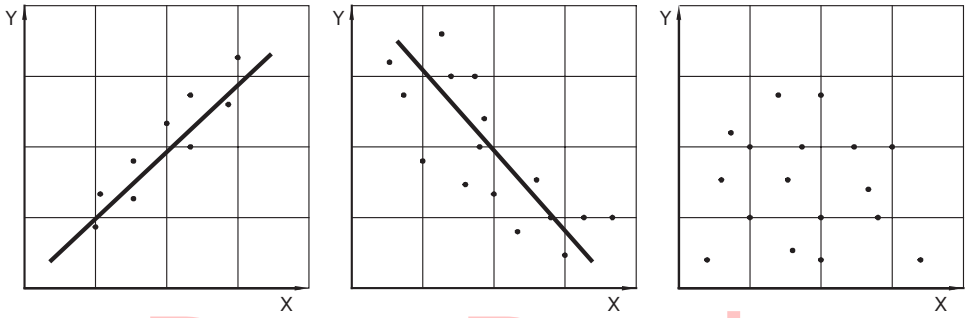

Figure 1.26 Cases of different correlation's

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To measure the strength of linear relationship between X and Y use the relation (1.179):

$$Y_i - \bar{Y} = \left(\widehat{Y}_i - \bar{Y}\right) + \left(Y_i - \widehat{Y}_i\right) \tag{1.215}$$

If there was a full and functional relationship between X and Y factors, then all Y_i experimental values would be equal to the values from regression $\widehat{Y}_i$ and all the data points in the scatter diagram would fall on the regression line. In such a case there would be no diversions of experimental values of a dependent variable from regression. Namely, the second member of the right-hand side of formula (1.215) would equal zero. Similarly, by analyzing the sum of squares versus Eq. (1.177) $SS_{TC}=SS_R+SS_E$ we would get $SS_E=0$, or a perfect description of experimental data by regression equation. The other extreme case is when there is no linear connection between variables. In that case all the values of the real regression $\widehat{Y}_i$ are equal to the arithmetic average of the dependent variable $\bar{Y}$, or the first number of the right-hand side of formula (1.215) would equal zero $SS_R=0$. The *coefficient of determination* is defined in accord with previous explanation r^2:

$$r^2 = \frac{SS_R}{SS_{TC}} = \frac{SS_{TC} - SS_E}{SS_{TC}} = 1 - \frac{SS_E}{SS_{TC}} \tag{1.216}$$

The coefficient of determination is that proportion of the total variability in the dependent variable that is accounted for by the regression equation in the independent variable(s). A value for r^2 of 1 indicates that the fitted regression equation accounts for all the variability of the values of the dependent variable in the sample data. At the other extreme, a value of 0 for r^2 indicates that the regression equation accounts for none of the variability. In other cases it has values between zero and one, and its value will therewith approach one if the linear relationship is stronger and zero if it is weaker. The coefficient of determination root square is called the *correlation coefficient* r.

According to all things said for the coefficient of determination, the correlation coefficient itself is a measure of the strength of relationship and it takes values between –1 and +1. When the correlation coefficient nears one the linear relationship between variables is strong, and when it is close to zero it means that there is no linear relationship between variables. This, however, does not mean that there is no relationship between variables, which might even be strong, of a certain curved shape. We point out that the correlation coefficient is an indefinite number, i.e. it does not depend on the units the variables have been expressed in.

The following is accepted as an empirical rule:

- correlation coefficient up to 0.30 indicates a weak relationship and is of uncertain validity;
- correlation coefficient between 0.50 and 0.70 indicates a significant relationship and is of practical importance;
- correlation coefficient above 0.90 means a strong relationship.

In statistical studies it is often more convenient to determine the correlation coefficient and then the regression equations.

1.7.1 Correlation in Linear Regression

For the simple linear regression model, $Y=\beta_0+\beta_1 X+\varepsilon$, the sum of squares due to regression is:

$$SS_R = b_1^2 \sum_i \left(X_i - \bar{X}\right)^2$$

Thus for the simple linear model we have:

$$r^2 = \frac{SS_R}{SS_{TC}} = \frac{b_1^2 \sum_i \left(X_i - \bar{X}\right)^2}{\sum_i \left(Y_i - \bar{Y}\right)^2} = \frac{\left[\sum_i \left(X_i - \bar{X}\right)\left(Y_i - \bar{Y}\right)\right]^2}{\sum \left(X_i - \bar{X}\right)^2 \sum \left(Y_i - \bar{Y}\right)^2} \tag{1.217}$$

since:

$$b_1 = \frac{\sum_i \left(X_i - \bar{X}\right)\left(Y_i - \bar{Y}\right)}{\sum \left(X_i - \bar{X}\right)^2} \quad \text{we have:} \quad r = \frac{\sum_i \left(X_i - \bar{X}\right)\left(Y_i - \bar{Y}\right)}{\left[\sum \left(X_i - \bar{X}\right)^2 \sum \left(Y_i - \bar{Y}\right)^2\right]^{1/2}} \tag{1.218}$$

Example 1.52

Referring to the data of Example 1.42 and using Eq. (1.218) we calculate the simple linear correlation coefficient as:

$$r^2 = \frac{167.5^2}{82.5 \times 342.1} = 0.994; r = \sqrt{0.994} = 0.996$$

indicating that the regression equation accounts for 99.4% of the variability of the data about $\bar{X}$. Since $\sum (X_i - \bar{X})(Y_i - \bar{Y})$=167.5, $r = \sqrt{0.994} = 0.996$. This means that X and Y are positively correlated. This means that as X increases or decreases the corresponding values of Y increase or decrease, accordingly. This also implies that the slope of the regression line is positive. In this example, the value of the correlation coefficient is quite high, r=0.996, indicating a "strong" linear relationship. Part of the variability 0.6% is not explained by the regression model and it is the consequence of not taking into account all the factors affecting the response variability, of not choosing the right form of a regression model, and of measurement errors. We have mentioned earlier that the square root of coefficient of determination gives the correlation coefficient r=0.996 a positive one as:

$$\sum_i (X_i - \bar{X})(Y_i - \bar{Y}) = +167.5$$

The positive correlation means: if X increases or decreases the corresponding values of Y increase or decrease too. The correlation coefficient may also be expressed by *covariance* of sample S_{XY} as:

$$b_1 = \frac{S_{XY}}{S_X^2} \tag{1.219}$$

so that:

$$r = \frac{S_{XY}}{S_X S_Y} \tag{1.220}$$

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Coefficient of determination-regression statistical significance-lack of fit of regression

It has already been mentioned that the coefficient of determination is that proportion of the total variability in the dependent variable that is accounted for by the regression equation in the independent variable(s). A value for r^2 of 1 indicates that the fitted regression equation accounts for all the variability of the values of the dependent variable in the sample data. At the other extreme, a value of 0 for r^2 indicates that the regression equation accounts for none of the variability.

A conclusion cannot, however, be drawn that the high value of the coefficient of determination simultaneously means a statistical significance of regression. In fact, one can obtain a value of 1 for r^2 by simply fitting a regression equation that includes as many (statistically estimable) terms as there are observations (i.e., data points). When the number of observations exceeds the number of terms in the regression equation by only a small number then the coefficient of determination might be large, even if there is no true relationship between the independent and

dependent variables. For example, the chances are one in ten of obtaining a value of r^2 as high as 0.9756 in fitting a simple linear regression equation to the relationship between an independent variable X and a normally distributed dependent variable Y based on only 3 observations, even if X is totally unrelated to Y, i.e., this result can occur 10% of the time, even if the two variables are unrelated. On the other hand, with 100 observations a coefficient of determination of 0.07 is sufficient to establish statistical significance of a linear regression at the 1% level. More generally, Table 1.81 indicates the value of r^2 required to establish statistical significance for a simple linear regression equation.

Table 1.81 Values of r^2 for a simple regression

Sample size	Statistical significance level		
	α=0.1	α=0.05	α=0.01
3	0.9756	0.9938	0.9998
4	0.810	0.9030	0.9800
5	0.65	0.77	0.92
6	0.53	0.66	0.84
7	0.45	0.57	0.77
8	0.39	0.50	0.70
9	0.34	0.44	0.64
10	0.03	0.40	0.59
11	0.27	0.36	0.54
12	0.25	0.33	0.50
13	0.23	0.31	0.47
14	0.21	0.28	0.44
15	0.19	0.26	0.41
20	0.14	0.20	0.31
25	0.11	0.16	0.26
30	0.09	0.13	0.22
40	0.07	0.10	0.16
50	0.05	0.08	0.13
100	0.03	0.04	0.07

Note that Table 1.81 applies only for a simple linear regression equation. For the case of multiple regression, statistical significance of the overall regression equation can be determined by the F-ratio in the analysis of variance [22]. Practical significance and statistical significance are not equivalent. With a small sample, it is possible not to obtain any evidence of a statistically significant regression relationship between two variables even if their true relationship is quite strong. This is because, as seen above, a relatively high value of r^2 is required to show a regression equation to be statistically significant when only a small number of observations are used. On the other hand, a regression equation based on only a modest (and practically unimportant) true relationship may be established as statistically significant if a suffi-

ciently large number of observations are available. For example, it was seen that with 100 observations a value for r^2=0.07 was sufficient to establish a highly significant statistical linear relationship between two variables.

Furthermore, the magnitude of r^2 depends directly on the range of variation of the independent variables for the given data. The coefficient of determination thus decreases with a decrease in the range of variation of the independent variables, assuming the correct regression model is being fitted to the data. For example, Fig. 1.27 shows the fitted regression equation between an independent variable, X, and a dependent variable, Y, based on 110 equally spaced values of X over the range from 10 to 20. The estimated coefficient of determination is r^2=0.89. However, if one had available only the 30 observations in the range 14 to 16 (see Fig. 1.28), the resulting coefficient of determination from the fitted regression equation would be only r^2=0.21.

Thus a large value of r^2 might reflect the fact that the data had been obtained over an unrealistically large range of variation. Conversely, a small value of r^2 might be due to the limited range of the independent variables. This is sometimes the case in analyzing data from a manufacturing process in which normal plant practice restricts the range of the process variables. Note also that a large and statistically significant coefficient of determination does not assure that the chosen regression model adequately represents the true relationship for all purposes. A coefficient of determination of r^2 =0.99, even if statistically significant , for a regression model involving only linear terms for each of independent variables, does not mean that a model that also includes quadratic and interaction terms could not conceivably yield a significantly better fit, nor that the "real cause variables" have been included in the regression equation.

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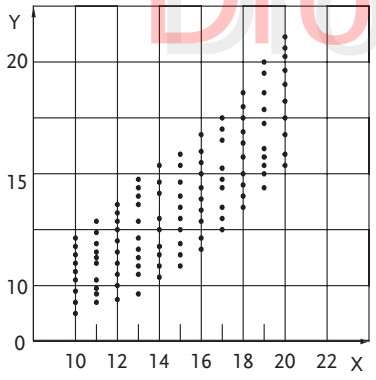

Figure 1.27 Data plots with $r^2 = 0.89$

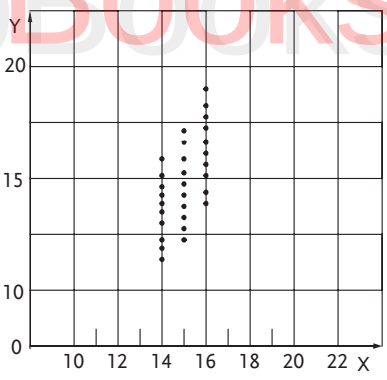

Figure 1.28 Data plots with $r^2 = 0.21$

1.7.2 Correlation in Multiple Linear Regression

In multiple linear regression where the model is:

$$Y=\beta_0+\beta_1X_1+\beta_2X_2+\ldots+\beta_pX_p+\varepsilon \tag{1.221}$$

the coefficient of determination is, according to Eq. (1.203):

$$r^2=\frac{SS_R}{SS_{TC}}=\frac{\left[b_1\sum_i\left(X_{1i}-\bar{X}_1\right)\left(Y_i-\bar{Y}\right)+\ldots+b_p\sum_i\left(X_{pi}-\bar{X}_p\right)\left(Y_i-\bar{Y}\right)\right]}{\sum_i\left(Y_i-\bar{Y}\right)} \tag{1.222}$$

Eq. (1.222) is analogous to (1.217). The coefficient r^2 as defined by Eq. (1.222) is called the *multiple coefficient of determination* or the *multiple correlation coefficient* r.

Example 1.53

The multiple coefficient of determination may be obtained for the data of Example 1.47 as a means of determining the "goodness of fit" of the regression equation already estimated. Eq. (1.222) is used to give:

$$r^2=\frac{b_1\sum\left(X_{1i}-\bar{X}_1\right)\left(Y_i-\bar{Y}\right)+b_2\sum\left(X_{2i}-\bar{X}_2\right)\left(Y_i-\bar{Y}\right)}{\sum\left(Y_i-\bar{Y}\right)}$$

therefore,

$r^2=(-0.12748\times10937.84+3.60271\times2959.17)/9274.97=0.9991;\qquad r=0.9995.$

Such a high correlation coefficient indicates that the regression model describes the experimental data extremely well. Apart from the mentioned multiple correlation coefficient the following *partial coefficient of determination*:

$r^2_{X_1X_2}=0.8712;\qquad r^2_{X_1Y}=0.8288;\qquad r^2_{X_2Y}=0.9956$

It is clear that from the partial coefficient of determination $r^2_{X_2Y}=0.9956$ and multiple coefficient of determination $r^2_{X_1X_2Y}=0.9991$ very little was gained by adding X_1 to the correlation.

Problem 1.49 [22]

A study was made on the effect of temperature on the yield of a chemical process. The following data (in coded form) were collected:

X: -5; -4; -3; -2; -1; 0; 1; 2; 3; 4; 5;
Y: 1; 5; 4; 7; 10; 8; 9; 13; 14; 13; 18;

Determine:

- Assuming a model, $Y=\beta_0+\beta_1X+\varepsilon$, what are the least squares estimates of regression coefficients.
- Do analysis of variance for significance level $\alpha=0.05$.
- What are the confidence limits for $_1$ ($\alpha=0.05$).

- What are the confidence limits (1-α =95%) for the true mean value of Y when X=3.
- What are the confidence limits (1-α =95%) for the true mean value of Y when are X=3 and X=-2.

Problem 1.50 [22]

Thirteen specimens of 90/10 Cu-Ni alloys, each with a specific iron content, were tested in a corrosion-wheel setup. The wheel was rotated in salt sea water at 30 ft/sec for 60 days. The corrosion was measured in weight loss in milligrams/square decimeter/day, *MDD*. The following data were collected:

X%Fe:	0.01;	0.48;	0.71;	0.95;	1.19;	0.01;	
Y loss in MDD:	127.6;	124.0;	110.8;	103.9;	101.5;	130.1;	
X%Fe:	0.48 ;	1.44;	0.71;	1.96;	0.01;	1.44;	1.96;
Y loss in MDD:	122.0;	92.3;	113.1;	83.7;	128.0;	91.4;	86.2.

Determine coefficients in the linear regression model and do analysis of variance taking into account check of lack of fit of the obtained regression model.

Problem 1.51 [24]

Two colorimetric methods were compared by measuring the contents of a chemical component. Based on experimental results determine whether there exists linear regression dependence between the method's.

Method I:	3720;	4328;	4655;	4818;	5545;	7278;	7880;	10085;	11707;
Method II:	5363;	6195;	6428;	6662;	7562;	9184;	10070;	12519;	13980.

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Problem 1.52 [24]

Temperature functions were mechanically tested for prepared sapphire samples. Find the linear regression dependence between the measured *Young's*-modulus and temperature.

X °C:	30;	100;	200;	300;	400;	500;	600;	700;	800;
Y:	4642;	4612;	4565;	4513;	4476;	4433;	4389;	4347;	4303;
X °C	900;	1000;	1100;	1200;	1300;	1400;	1500;		
Y:	4251;	4201;	4140;	4100;	4073;	4024;	3999.		

Problem 1.53 [10]

Two procedures were tested in developing a method for measuring blood flow. Based on obtained results determine whether there exists linear correlation between the procedures, and if there is, give the linear regression analysis of variance.

X:	1190;	1455;	1550;	1730;	1745;	1770;	1900;	1920;	1960.
Y:	1115;	1425;	1515;	1795;	1715;	1710;	1830;	1920;	1970.
X:	2295;	2335;	2490;	2720;	2710;	2530;	2900;	2760;	3010.
Y:	2300;	2280;	2520;	2630;	2740;	2390;	2800;	2630;	2970.

Problem 1.54

Moisture content in the mixture of a product has an influence on density of the final product. The moisture of the analyzed mixture has been controlled and the density of the final product measured. Experimental values offered:

X%:	4.7;	5.0;	5.2;	5.2;	5.9;	4.7;	5.9;	5.2;	5.3;	5.9;	5.6;	5.0;
Yg/cm^3:	3;	3;	4;	5;	10;	2;	9;	3;	7;	6;	6;	4.

Determine:

a) linear regression model $Y=\beta_0+\beta_1 X+\varepsilon$;
b) 95% confidence interval for β_1 ;
c) analysis of variance and check lack of fit of the model.

Problem 1.55 [4]

The relation between the heat capacity of liquid sulfuric acid in cal/g °C and temperature in °C is as follows:

Ccal/g °C:	0.377;	0.389;	0.396;	0.405;	0.466;	0.458;
T °C:	50;	100;	150;	200;	250;	300

Determine regression coefficients in the linear regression: $C_p=\beta_0+\beta_1 T+\varepsilon$.

Problem 1.56 [4]

The irritant factor Y of polluted air can be determined as a function of the concentrations of SO_2 and NO_2 in the atmosphere. The following data are available where X_1 = parts NO_2 per ten million parts of air and X_2 = parts SO_2 per hundred million parts of air. Determine the irritant factor as a function of X_1 and X_2:

X_1:	10;	12;	15;	16;	19;	21;	25;	28;
X_2:	12.5;	15;	18;	21;	26;	30;	35;	40;
Y:	65;	72;	82;	95;	110;	122;	125	130.

Problem 1.57

In the production of ethylene glycol from ethylene oxide, the conversion of ethylene to ethylene oxide, X, is a function of the activity Z_1 of the silver catalyst and the residence time Z_2 . the following coded data are available:

X:	12.1;	11.9;	10.2;	8.0;	7.7;	5.3;	7.9;	7.8;	5.5;	2.6;
Z_1:	0;	1;	2;	3;	4;	5;	6;	7;	8;	9;
Z_2:	7;	4;	4;	6;	4;	2;	1;	1;	1;	0.

a) Write a suitable model;
b) What portion of the data does your regression equation explain?

Problem 1.58 [4]

Two supposedly identical Brook's-model R-2-65-5 rotameters with 316 stainless-steel spherical floats were calibrated for helium service at 20 psig input, 74 °F . Let Y mlHe/min. =flow rate and X mm=scale reading. The data are below:

X:	10;	10;	14;	16;	19;	20;	25;	30;	30;	35;
Y_1 :	9.2;	9.5;	-;	15.0;	-;	21.3;	29.4;	41.0;	40.5;	54.0;
Y_2 :	8.4;	8.6;	12.6;	-;	18.6;	18.0;	27.0;	36.8;	36.3;	49.1.

X:	35;	40;	40;	45;	45;	50;	50;	55;	55;	60;
Y_1:	55.0;	68.0;	68.8;	86.0;	88.1;	103.2;	104.6;	124.0;	123.1;	144.0
Y_2:	50.0;	64.8;	64.8;	81.3;	82.0;	100.4;	97.0;	114.7;	117.0;	134.0

Problem 1.59 [25]

Magnetic material is mechanically separated from the slurry of ground ore and rolled into balls that are to be sent to furnaces for producing small balls. To reinforce the material better and to give it greater hardness a binder such as natural peat is usually added. The content of the binder has an effect on ball hardness, as can be seen from experimental values.

Y:	3.6;	9.8;	14.7;	16.2;	16.0;	15.5.
X:	0.0;	4.0;	8.0;	12.0;	16.0;	20.0.

Determine the relationship between grinding and content of the binder.

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Problem 1.60

The temperature effect on bleaching of a final product was determined experimentally. The obtained data are:

X K:	460;	450;	440;	430;	420;	410;	450;	440;
Y bleaching degree:	0.3;	0.3;	0.4;	0.4;	0.6;	0.5;	0.5;	0.6;
X K:	430;	420;	410;	400;	420;	410;	400	
Y bleaching degree:	0.6;	0.6;	0.7;	0.6;	0.6;	0.6;	0.6;	

Determine:

a) Linear regression model $Y=\beta_0+\beta_1 X+\varepsilon$.
b) Analysis of variance and check lack of fit of the regression model.
c) 95% confidence interval for the mean Y for X values.

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II
Design and Analysis of Experiments

2.0
Introduction to Design of Experiments (DOE)

Design of experiments, like any other scientific discipline, has its own terminology, methodology and subject of research. The title of this scientific discipline itself clearly indicates that it deals with experimental methods. A large number of experiments is done in research, development and optimization of the system. This research is done in labs, pilot plants, full-scale plants, agricultural lots, clinics, etc. An experiment may be physical, psychological or model based. It may be performed directly on the subject or on its model. The model usually differs from the subject in its dimensions and sometimes in its nature. The experiment may also be done on an abstract mathematical model. When a model describes the subject precisely enough, the experiment on the subject is generally replaced by an experiment on the model. Lately, due to a rapid development of computer technology, physical models are more frequently replaced by abstract mathematical ones.

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An experiment takes a central place in science, particularly nowadays, due to the complexity of problems science deals with. The question of efficiency of using an experiment is therefore imposed. *J. Bernal* has made an estimation that scientific research is organized and done fairly chaotically so that the coefficient of its usability is about 2%. To increase research efficiency, it is necessary to introduce something completely new into classical experimental research.

One kind of innovation could be, to apply statistical mathematical methods or to develop *design of experiments-DOE. DOE is a planned approach for determiniing cause and effect relationships.*

Hereby, the following is essential:

- reduction or minimization of total number of trials;
- simultaneous varying of all factors that formalizes experimenter's activities;
- choice of a clear strategy that enables reliable solutions to be obtained after each sequence of experiments.

The methodology of design of experiments has in developed countries made a special expansion in solving very complex problems in all fields of human activities. It should be pointed out that an important place in this expansion was the develop-

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ment of electronic computers, for they greatly accelerated and alleviated statistical calculations.

Chemical and engineering studies, as for those in other fields, are based on complex, long-term and relatively expensive experiments. Experimental work is included in:

- physical and chemical studies for establishing constants and properties of elements, chemical compounds and materials;
- routine analyses of raw materials, intermediates and final products;
- lab studies for designing and developing technological processes;
- optimization of technological procedures in the lab, pilot-plant and full-scale plant systems;
- optimization of mixture or *"composition-properties"*;
- mathematical modeling of a system;
- selection of factors by the significance of their effects on a measured value-response;
- estimates and definitions of theoretic model constants, etc.

Hence, wherever experiments exist there should be new scientific disciplines dealing with their designing and analysis.

The efficiency of experimental research is determined by the degree of precision and completeness of data and information about the system that is being tested. This degree results from applying the methodology of design on the experiments and on the way the obtained experimental data are analyzed. It is important at this point to consider the manner in which the experimental data were collected as this greatly influences the choice of the proper technique for data analysis. Before going any further it is well to point out that the person performing the data analysis should be fully aware of several things:

- What is the objective of the research?
- What is considered a significant research finding?
- How are the data to be collected and what are the factors that effect the responses?

If an experiment has been properly designed or planned, the data will be collected in the most efficient form for the problem being considered. Experimental design is the sequence of steps initially taken to insure that the data will be obtained in such a way that its analysis will lead immediately to valid statistical inferences. Before a design can be chosen, the following questions must be answered:

- How to measure the response and the factor's effect?
- How many of the factors will affect the response?
- How many of the factors will be considered simultaneously?
- How many replications (repetitions) of the experiment will be required?
- What type of data analysis is required (regression, ANOVA, etc.)?
- What level of difference in effects is considered significant?

The purpose of statistically designing an experiment is to collect the maximum amount of relevant information with a minimum expenditure of time and resources. It is important to remember also that the design of experiment should be as simple as possible and consistent with the requirements of the problem. Hence, design of experiments requires a new approach to research, which is far from the traditional (classical) methods of empirical research. The traditional approach demands considerable material expense and is more time consuming, for the effect of each factor experiment may be designed to investigate one factor at a time so that all other independent variables (factors) are held constant. This is the so-called classical experimental design and is the one that has been favored almost exclusively among scientists and engineers. At the same time, the factors have no more than 4 or 5 different values (levels of variation) as the total number of trials is particularly big. If, for instance, the effect of five factors is to be tested where each of them may be varied at five levels, then for the complete testing of the research subject it is necessary to realize 5^5=3125 different combinations of factors-trials with no trial replications meant to reduce experimental errors. The plotted number of classical experimental design points is hard to realize, so that in practice their number is reduced at the expense of either reducing the investigated factor space-domain or the number of factor levels. In both cases, the confidence of conclusions, based on experimental results, is reduced.

Besides, a significant part of information obtained in a similar way is of no practical use for it refers to the region of factor space-domain, which is far from its optimum. Even more drastic errors are possible if all the necessary trials are done. However, due to the huge time consumption, uncontrolled changes in the quality of inlet raw materials or in the experimental plant are not accounted for. The first and final trial results of an experimental program are not comparable from the accuracy point of view. As an important drawback of classical experimenting, there also appears the fact that it is impossible to single out the effects of interactions between the analyzed factors. This has a great influence on the errors in estimating the responses as functions of observed factors. An additional difficulty also arises in an estimate on the lack of fit of the obtained mathematical model since the experimental error is usually missing. Finally, interpreting the results of a classical experiment becomes difficult, because a simultaneous analysis is impossible due to a large number of tables and graphs.

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Most of these problems can be avoided by applying the design of experiments and a simultaneous increase in efficiency of empirical research. The consumption of research time may be reduced ten or more times. Referring to the example where five factors are analyzed, it is possible to do the designed experiment with 32 trials only by using rotatable design of second order. Cases are known when, by applying the design of experiments, an optimal solution has been reached and where a classical experiment had no solution in a reasonable time period.

By using the design of experiments, a researcher's intuition is developed and his way of thinking changed. It may therefore be said, that the design and analysis of an experiment is a scientific method in elaborating experimental results, in finding optimal solutions and in research that has the experiment as their subject. Design

of experiments also uses a traditional approach in the research, namely the use of experimental data to obtain a mathematical model of a system. In a general case and from a mathematical point of view, the used mathematical models may end up being complicated mathematical functions.

Response, aim function or *optimization criterion* may have the form:

$$y=f(x_i, z_i, w_i) \tag{2.1}$$

where:
y is response, aim function, optimization criterion;
x_i are the controllable independent variables, factors;
z_i,w_i are variables and constants that affect y but are uncontrollable;
f is the function that defines y, x_i, z_i, w_i relationships.

Besides, one should also keep in mind the equations and non-equations that define the constraints of controllable factors. Equation (2.1) defines the constraints of a research subject. Research solutions may be considered optimal if they are the maximum and minimum of the response function for the given constraints.

It has to be remembered that each model is an approximate solution and generally is not a correct description of the research subject. Optimal solution of a model is therefore considered an approximate optimum of the real system. This assertion is both good and bad. The good side is that the models are not complicated, since, to be close to the real system, they would have to be very complex. On the other hand, insufficient reality of a model reduces the solution confidence.

In classical research methods, the main objective is to define the rule/law, which has the property of an absolute category, at a given level of knowledge. The law is either unconditionally correct or not. Such an approach makes studying a complex system difficult, for when many factors have complex effects it is difficult to find the correct mathematical system in accord with the laws. Also, approximate solutions are senseless for we cannot talk about "*bad*" and "*good*" laws. In the new approach to solving problems, or in design of experiments, the mathematical model is not absolute. It only offers an approximate idea on the research subject and one may speak of "good" and "bad" mathematical models. The essence of design of experiments is that it enables optimal solutions to be obtained even when it is really impossible to get a functional (deterministic) mathematical model and define a rule precisely. It is characteristic for design of experiments that it uses polynomial models since the quality of approximation may be improved by increasing a polynomial degree. Such models are especially suitable for solving optimization problems as it makes it possible to take into account the effects of interaction and a large number of factors. Besides, it makes it easy to estimate the degree of lack of fit of polynomial models of different orders.

A designed or active experiment is based on using general methodological concepts such as regression and correlation analysis, analysis of variance, randomization, optimal use of factor space, successive experimenting, replication, compactness of information, statistical estimates, etc.

The regression analysis mathematical apparatus is used in the design of experiments. It is therefore suggested to take into account assumptions of regression anal-

ysis when performing an experiment. This means that the trial results are independently and normally distributed random values of equal variances. In other words, the experimental results in each trial are obtained with certain probability so that the distribution of such values in each trial is subject to the normal distribution law, and variances typical for them are practically equal. The law on the distribution of experiment results is observed because, the random value is defined if its distribution law is known. The stress is on the normal distribution for then the used mathematical model is the most efficient. The law on normal distribution of data is most frequently met in practice. The fact that some experimental results do not submit to this law is not upsetting as by mathematical transformations, given in section 1.5, such results may be brought down to the normal distribution law. Equality of random-value variances is of particular importance in experiments with a minimal number of runs or design of experiments due to their confidence level. This condition is fulfilled if the variance of one trial is equal to the same variance of any other trial. This variance equality is checked by tests from section 1.5. In the case of inequality, it is solved by identical transformations, same as for the normality of data distribution. These checks may be easily performed since replication of trials is available and replicated trials are a principle of design of experiments.

One assumption of regression analysis is the increased precision of measuring or fixing a factor. When measuring or fixing a factor, such conditions are recommended where a factor measurement error is incomparably smaller when compared to an error in determining a response.

Randomization is also an important idea in the design of experiments. It has to do with the random sequence of doing trials so as to annul the influence of systematic factors, which are difficult to stabilize and control. In this way one of the main concepts of classical experiment, having to do with the necessity of fixing disturbance factors, is disrupted. Randomization is the means used to eliminate any bias in the experimental units and/or treatment of combinations-trials. If the data are random it is safe to assume that they are independently distributed. Errors associated with experimental units, which are adjacent in time or space will tend to be correlated, thus violating the assumption of independence. Randomization helps to make this correlation as small as possible so that the analyses can be carried out as though the assumption of independence were true.

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The idea of the concept of successiveness in doing an experiment is as follows. Empirical research should consist of separate successive stages or series of trials and not of designing a complete experimental research in advance. An active experiment should have the property of successiveness, or, each next stage is projected and designed based on the results of previous trials.

Optimality of using the factor space for an adequate multifactor experiment means an increase in experiment efficiency proportional to the increase in the number of its factors.

The estimate precision of a polynomial model regression coefficients rises with an increase in the number of factors, because the diameter of the sphere of factor space, within which variation limits of each factor lie, also increases.

The concept of information compactness refers to the result analysis of a designed experiment. This means that final results do not require a large number of tables and graphs.

The concept of statistical estimates refers to the threshold or significance level where the estimate of a parameter, model or solution is either accepted or rejected.

Finally, it should be pointed out once again that obtaining as precise and complete information on a studied chemical or physical system as possible, with a minimal number of experiments and the lowest possible expenses, is the necessary condition for efficient research work. Therefore, application of modern mathematical and statistical methods in designing and analyzing experimental results is a real necessity in all fields and phases of work, starting with purely theoretical considerations of a process, its research and development, all the way to designing equipment and studying optimal operational conditions of a plant.

All empirical research methodologies may be divided into two large groups:

- classical or passive,
- active or statistically designed.

Classical design of experiments-one factor at a time

Experiments may be designed to investigate one factor at a time so that all other independent variable-factors are held constant. This is the so-called classical experimental design. A classical experiment means researching mutual relationships between variables of a system, under *"specially adapted conditions"*.

Let us observe an example of system research where the effects of k factors on p levels are to be determined. As we mention above, the classical system of experimenting requires each factor to be tested at p levels while others are kept constant at chosen fixed values. The total number of trials to be done by this scheme is:

$$N=k(p-1)+1 \tag{2.2}$$

Assume we have the production in a chemical reactor whereby the product yield y is essentially affected by three factors: X_1 reaction mixture temperature, X_2 pressure in reactor and X_3 time of reaction. If all factors are changed at two levels (p=2) then the research program is encompassed by four trials (N=4). The lower level factor values are marked by the symbol "-" and the upper ones by "+". The conditions of doing each run are shown in Table 2.1.

Table 2.1 Experimental combinations

Number of trials	Factor level combinations			y	Remark
	X_1	X_2	X_3		
1	−	−	−	y_1	Reference run
2	+	−	−	y_2	
3	−	+	−	y_3	
4	−	−	+	y_4	

After realizing each trial, it is possible to determine factor effects on product yields:

$E_{X1}=y_2-y_1$; temperature effect on yield;

$E_{X2}=y_3-y_1$; pressure effect on yield; (2.3)

$E_{X3}=y_4-y_1$; time effect on yield;

Based on data analysis one can conclude that:

- lack of experimental error;
- lack of interaction effects;
- the result of referential trial (y_1) is overestimated for it is used three times in determining the effects.

Based on this kind of analysis the researcher may decide to check the precision of the results by repeating the trials. Precision is the repeatability of the results of a particular experiment. However, apart from the possibility of determining experimental error, the trial repeating does not offer new information.

Statistical design of experiments-DOE

The mentioned deficiencies of the classical design of an experiment may efficiently be removed and overcome by statistical design and calculation of obtained results by means of methods of statistical analysis.

If for the studied example, instead of repetition, the experimental program is expanded by additional combinations of factor levels-trials, as shown in Table 2.2, we get an experiment with eight trials.

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Table 2.2 Additional experimental combinations

Number of trials	Factor level combinations			y	Remark
	X_1	X_2	X_3		
5	+	+	−	y_5	
6	+	−	+	y_6	
7	−	+	+	y_7	
8	+	+	+	y_8	

A complete design of experimental research, which includes all eight design points, is one of the best-known statistical experimental designs, the so-called *full factorial design*.

Factorial design of experiments, combined with statistical methods of data analysis, offers wider and more differentiated information on the system, while conclusions are of greater usability. The results of all the eight runs in the analyzed example serve for determining the factor effects, with seven trials being independent possibilities of testing the effects and one serving for their comparison with the chosen fixed values. Three out of seven independently determined factor effects serve for

finding its basic effect: E_{X1}; E_{X2} and E_{X3} and the other four to determine their mutual interactions: E_{X1X2} E_{X1X3} E_{X2X3} and E_{X1X2X3}, following these expressions:

Table 2.3 Full factorial design2^k

Number of trials	Factor level combinations			Response y	Remark
	X_1	X_2	X_3		
1	−	−	−	y_1	Reference trial
2	+	−	−	y_2	
3	−	+	−	y_3	
4	+	+	−	y_4	
5	−	−	+	y_5	
6	+	−	+	y_6	
7	−	+	+	y_7	
8	+	+	+	y_8	

$$
\begin{aligned}
E_{X1} &= (y_2+y_4+y_6+y_8)/4-(y_1+y_3+y_5+y_7)/4\\
E_{X2} &= (y_3+y_4+y_7+y_8)/4-(y_1+y_2+y_5+y_6)/4\\
E_{X3} &= (y_5+y_6+y_7+y_8)/4-(y_1+y_2+y_3+y_4)/4\\
E_{X1X2} &= (y_1+y_4+y_5+y_8)/4-(y_2+y_3+y_6+y_7)/4\\
E_{X2X3} &= (y_1+y_2+y_7+y_8)/4-(y_3+y_4+y_5+y_6)/4\\
E_{X1X3} &= (y_1+y_3+y_6+y_8)/4-(y_2+y_4+y_5+y_7)/4\\
E_{X1X2X3} &= (y_2+y_3+y_5+y_8)/4-(y_1+y_4+y_6+y_7)/4
\end{aligned}
\qquad (2.4)
$$

As has been said before, in the case of the classical experiment, which with replication has N=2×4=8 trials, the results of three trials are used for establishing basic factorial effects, one as a referential value and the remaining four for determining experimental error. The advantages of factorial design are evident and they prove to be the best in experiments with a larger number of factors. Basic advantages of *design of experiment* when compared to the one factor at a time classical one, are as follows:

- it makes possible asserting lawfulness of phenomena in the experimental space-domain as a whole, and hence drawing conclusion on results is of wider usability value;
- it offers wider possibilities of testing, the effects of factor varying on final result, since results of all trials are used for calculation of the effects;
- it enables establishing the size of factor interactions, moreover, this is the only way such interactions may be determined;
- data accuracy from an active experiment is reached through considerably fewer statistically designed trials, i.e. at the same number of trials an active experiment offers more complete and precise information;
- the final research objective set up is achieved in a systematic, well thought out and organized way in a short time with considerably fewer runs and the lowest possible material costs;

- in a classical experiment one is usually unable to take into account uncontrolled changes, errors resulting from material variation, bias errors and errors resulting from the sequence of testing;
- a classical experiment has a lack of information about experimental error, which serves as an estimate of the lack of fit for the obtained mathematical model;
- when doing a classical experiment one obtains clumsy tables and graphs that are difficult for a simultaneous analysis;
- an active experiment eliminates one of the main assumptions of classical experimentation having to do with the necessity of fixing disturbance factors. A researcher is consciously suggested to make random situations-randomizations so that hard to stabilize and uncontrolled factors could have a random character;
- an active experiment has a successive property, or, each next stage is projected and designed based on results of a previous series of trials;
- an active experiment changes the way of the experimenter's reasoning, increases his intuition and makes him active in projecting further stages of an experiment, requires use of empirical and scientific background;
- a classical experiment is a special case of an active statistical design of experiment where the individual effect of certain factors on system response is tested. From a mathematical point of view, a classical experiment offers partial effect, while the active one gives the total effect, for with it all factors are simultaneously varying in the experiment.

Table 2.4 shows basic statistical designs for all kinds of quantitative and categorical/qualitative factors.

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Table 2.4 Basic DOE Designs

Experimental design	Factors	Application
Simple comparative designs	Categorical/qualitative and quantitative	Check of method, testing of single factor effect
Random blocks and Latin squares	Differences between batches, treatments, samples	Calculation of effects with elimination of inequality of experimental conditions
Fractional replicate designs	Categorical/qualitative and quantitative	Screening of factors
Random balance design	Categorical/qualitative and quantitative	Screening of factors
Full factorial designs	Categorical/qualitative, quantitative and combined	Choice of factors, calculation of main effects and interactions
Central composite rotatable designs	Quantitative	Regression models of second order
Central composite orthogonal designs	Quantitative	Regression models of second order

Table 2.4 Continued

Experimental design	Factors	Application
Simplex lattice design	Quantitative	Mixture problems, regression models of second and higher orders
Extreme vertex design	Quantitative with constraints	Mixture problems, regression models of second and higher orders
Hartley's, Kono's, Kifer's, D-Optimal	Quantitative	Regression models
Higher-order designs	Quantitative	Regression models of higher order

2.1 Preliminary Examination of Subject of Research

2.1.1 Defining Research Problem

Experimental research of the system must be preceded by *preliminary examination of the subject of research* aimed at obtaining information necessary for defining the research objective.

The modern approach to experimental research presupposes that to obtain the optimal solution it is necessary to define the research problem correctly. It should be defined in such a way to enable the most efficient algorithms and methods of a designed experiment. For a concrete definition of a research problem, it is necessary to formulate clearly its objective, choose the research subject model and analyze its preliminary information. Special attention should be paid to the setup conditions in the problem with reference to the capability of the available experimental plant. The next step is the choice of *preliminary design of experiment*. When choosing it one must take into account all the singularities of the research problem and all known design of experiments must be analyzed in this respect. The design or method that is most efficient in the particular analyzed case is chosen. The methods and designs of experiments for further research stages will be considered after completing and analyzing the previous research. As Fig. 2.1 shows, the new approach to experimental research requires long prior preparation of the experiment aimed at increasing experimentation efficiency.

The research objective may be defined if the research subject or optimization subject is defined, if its requirements are known and if there exist interactions that change the quality of a research subject with the change of requirements.

The next step is choice of research subject model. It has been said before that design of experiments rests on cybernetic concepts about the research subject. A *"black box»* is therefore recommended as the research subject model, which will be affected by various controllable factors. The defining principles of such a model cor-

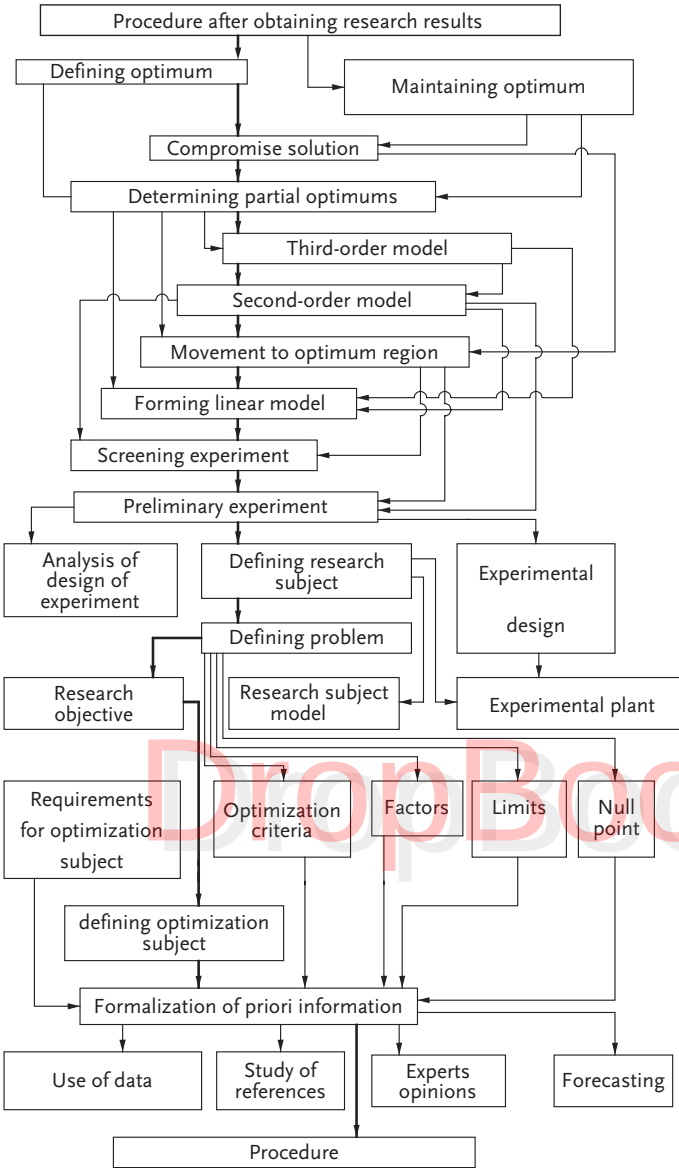

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Figure 2.1 Block diagram of experimental research

respond to the researcher's preliminary knowledge on insufficient awareness of the mechanism of multifactor research-problem phenomenon.

Figure 2.2 shows the black-box model. The *inlets* indicated by arrows X_1, X_2,..., X_k are the possibilities of affecting the research subject. The *outlet* arrows y_1, y_2,..., y_m or outlets are *responses, optimization criteria* or *aim functions*.

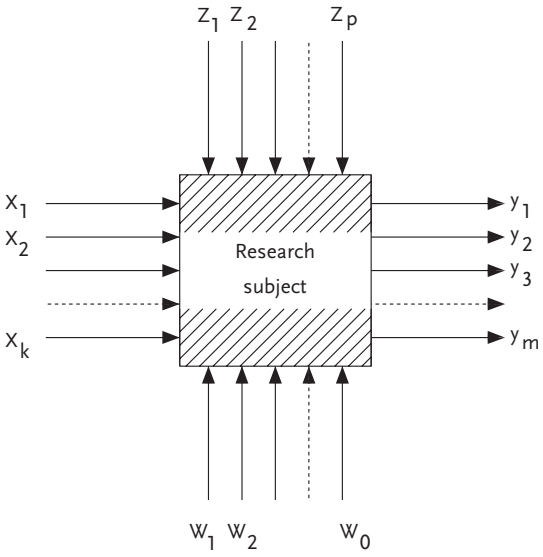

Figure 2.2 Black box model

Input variables are *controllable, uncontrollable* and *disturbance* variables. Controllable variables or *factors* $X_1, X_2,..., X_k$ are variables, that can be directed or that can affect the research subject in order to change the response. They can be numerical (example: temperature) or categorical (example: raw material supplier). Uncontrollable variables $Z_1, Z_2,..., Z_p$ are measured and controlled during the experiment but they cannot be changed at our wish. They can be a major cause for variability in the responses. Other sources of variability are deviations around the set points of the controllable factors, plus sampling and measurement error. Furthermore, the system itself may be composed of parts that also exhibit variability. Disturbance, non controlled variables $W_1, W_2,..., W_Q$ are immeasurable and their values are randomly changed in time.

Factors may have associated values called *levels* of variations. Each state of a black box has a definite combination of factor levels. The more different states of the black box that exist, the more complex is the research subject. Formalization of preliminary information includes: analysis of reference data, expert opinions and use of direct data, which enables correct selection of response, factors and *null point* or *center of experiment*. Factor limitations are also defined at this stage. If the research is linked with several following responses, then response limitations also have to be analyzed. The next phase refers to defining the research problem. When defining this problem one must keep in mind the research-subject model, and in a general case it is Eq. (2.1) that defines the link between the inlet and outlet of the black box. Defining the research problem is possible only now when its aim has been determined, the criteria established, the factors, limitations and null point defined. The problem is a simple one when only one response or optimization criterion is in

question. In the case of several optimization criteria or *multiple response optimization* the problem becomes very complex.

Defining a research objective by its difficulty may be divided into three levels:

- screening factors regarding statistical significance of their effect on response;
- obtaining a mathematical model of research subject;
- optimization of the research subject.

Optimization of a research subject is the hardest research problem. It should immediately be noted that different optimization problems appear in practice. In most cases *extreme problems* are present, problems of searching for extremes (minima and maxima) of a response function in the case of one response and with factor limitations. Most such problems have to do with finding the maxima of outlet and minima of inlet parameters. There are situations too where response improvement with regard to initial state in null point is required. Often, there is a demand for finding the local optimum if there are more of these.

Finding the mathematical model of the research subject is the lower level of a research objective. It is obligatory for a large number of problems. This obligation comes after the end of factor screening or after finding the optimum. The general form of the research subject mathematical form is:

$$y=\varphi(X_1,X_2,\ldots,X_k) \tag{2.5}$$

where:
y is response, optimization criterion, value that is measured during the experiment;
X_1, X_2,..., X_k-are controllable factors that are changed during the experiment.

The aim function may in this case be called *response function* for it is literally the response to factor change. Geometrically, a *response surface* corresponds to a response function.

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It has been said before that we use polynomial models in the design of experiments. Therewith we, in principle, approximate the response function *(2.5)* by a polynomial.

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i,j=1}^{k} \beta_{ij} X_i X_j + \sum_{i=1}^{k} \beta_{ii} X_{ii}^2 + \ldots \tag{2.6}$$

where:
β_0, β_i, βij, βii are theoretical regression or polynomial coefficients.

Based on experimental values, the real regression coefficients are estimated, so that:

$$\hat{y} = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i,j=1}^{k} b_{ij} X_i X_j + \sum_{i=1}^{k} b_{ii} X_{ii}^2 + \ldots \tag{2.7}$$

where:
$\hat{y}$ is predicted-calculated response value,
b_0, b_i, b_{ij}, b_{ii} are real regression coefficients.

From regression coefficient values one may estimate the factor effects or the degree of influence of associated factors on response. Geometrically, Eq. (2.7) is the

response surface in the k-dimensional region. This basic surface may, for a more detailed study of the optimum region, be cut two-dimensionally for constant response values, with the idea of obtaining a *contour graph*, which can easily be presented geometrically in a plane.

Lack of fit of the obtained model has to be statistically checked, so that, if needed, the polynomial degree may be augmented. Knowing the mathematical model of the research subject for several responses is a prerequisite in solving *optimization with multiple responses*. The computation of this is solved geometrically or by use of computers and the method of linear algebra.

2.1.2 Selection of the Responses

Selection of the responses is one of the most important problems of a preliminary study of the research subject, since a correct definition of research objective means correct selection of the responses. An incorrect selection of the responses annuls all further research activities. Depending on the subject and research objective, optimization parameters or responses may be quite different. To formalize the procedure of selection of the responses, with no intention of being detailed and complete, Fig. 2.3 gives the block diagram of the most frequently used optimization parameters.

This block diagram includes the most frequently used responses in practice and it can help the researcher to find his way in a real situation. Real situations are by rule very complex and usually require simultaneous analysis of several system responses. Each research subject may, in principle, be characterized by a population or any

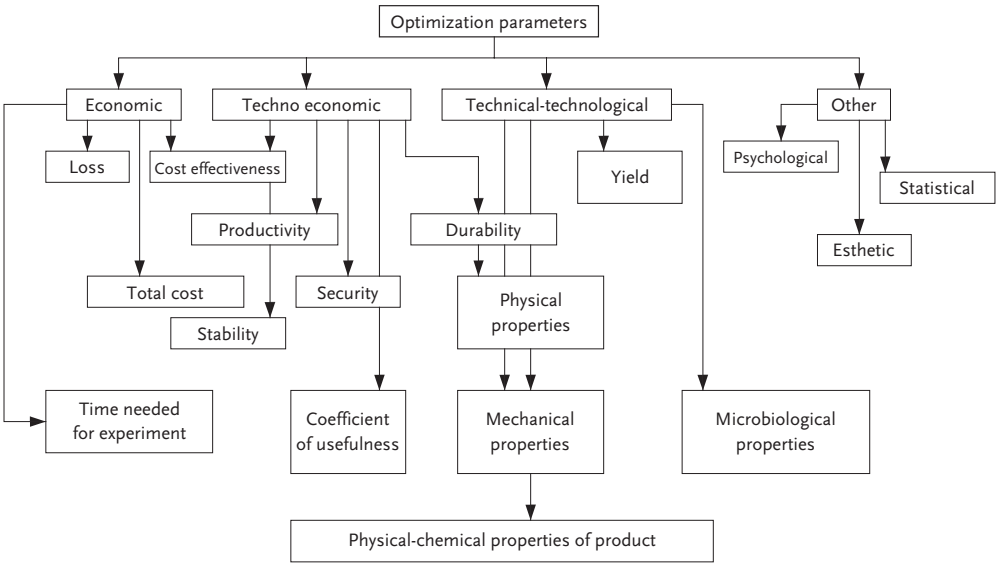

Figure 2.3 Block diagram of response selection

other response sub population, given in *Fig. 2.3*. Optimization of such a research subject may be done only when a unique optimization parameter has been selected. In such a case, all other responses are not optimization parameters but are taken as constraints. The other way is to make one, so-called *general response,* from all analyzed responses.

For a research subject parameter to be a response, it has to fulfill certain conditions. A response should be:

- quantitative,
- singular,
- statistically effective,
- universal,
- physically realistic,
- simple,
- easily measurable

System response should be quantitative, i.e. its property must be its ability to be expressed by ciphers. Its manner of measurement in any combination of factors, which determine it, must also be known. The sum of values taken by a response is called the *domain of response*. The domain of optimization parameter determination may be *continuous* and *discrete, limited* and *unlimited*. A chemical reaction yield, for example, is a continuous, limited response for it changes continually in a limited range from 0 to 100%. The number of rejected products and the number of plant damages are examples of discrete and (on one side) limited regions of response determination. When it is impossible to determine a response quantitatively, we use the method of *ranking*. By this method, definite estimates or ranks are corresponded to an optimization parameter by a predetermined defined scale. The ranked response obtained has a discrete limited determination region. A rank is a quantitative response estimate with a definite degree of subjectivity, i.e. it is associated with qualitative response meanings. For any physically measurable response it is possible to make up a response with ranks. Thereby one has to keep in mind the fact that the rank method gives a less sensitive response, which makes studying finer effects impossible.

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Singularity of response is such a property of a quantitative parameter where one and only one response value, with precision up to the size of experimental error, corresponds to a definite factor combination. It is obvious that the opposite is not valid, for several factor combinations may correspond to one response value.

Besides the two mentioned properties, an optimization parameter should also be statistically effective. This response property is brought down to the choice of optimization parameter with the highest possible precision of determination. When this response precision is insufficient, the number of trials is increased.

The universality of optimization parameter means a many-sided and total characterization of a research subject. With regards to universality, technological optimization parameters are not universal enough for they do not include a property such as cost efficiency of a process. General optimization parameters have universality, as they are a function of the necessary number of individual properties.

Another desire for an optimization parameter is also to have physical sense, to be simple and easy to measure. The physical sense of a response has to do with result interpretation, and simplicity and ease of measurement when doing an experiment.

With regards to the research subject as a system, which may consist of several subsystems, it should be kept in mind that in that case we can talk about *local* optimization parameters. Discovering local optima often does not mean that we have the optimum for the whole system.

Apart from the analyzed requirements to be fulfilled by an optimization parameter, one should also, when choosing the response, keep in mind the fact that this parameter affects, up to a point, the choice of the research subject model. Economic parameters are by their nature additive, so that they can be easily modeled by simple functions, which is not applicable to physical and chemical responses.

2.1.2.1 Subject of Research with Several Responses

Research problems with one response undoubtedly have an advantage. In practice, however, we mostly meet research subjects with several responses, which often means a literally large number of responses. Thus, for example, when producing rubber, plastic and other composite materials one must take into account responses such as: physical-chemical, technological, economic, mechanical (tensile strength, elongation, module, etc.) and others. One can define the mathematical model for each of the mentioned responses but simultaneous optimization of several functions is mathematically impossible.

In such cases we usually do the optimization by one response, which by the definition of the research objective, is the most important, while for others we impose constraints. A useful thing in such situations is to find a possibility of reducing the number of responses. This is where correlation analysis comes in. By means of correlation analysis one should determine correlation-coefficient pairs between all possible responses.

If one response is marked y_1, and the other one y_2 and if the number of runs they are measured in is N, then the correlation coefficient in case of u=1, 2,..., N number of trials, is given by expression:

$$r_{y_1 y_2} = \left[\sum_{u=1}^{N} (y_{1u} - \bar{y}_1)(y_{2u} - \bar{y}_2)\right] \Bigg/ \left[\sum_{u=1}^{N} (y_{1u} - \bar{y}_1)^2 (y_{2u} - \bar{y}_2)^2\right]^{1/2} \tag{2.8}$$

where:

$$\bar{y}_1 = \sum_{u=1}^{N} y_{1u}/N \quad \bar{y}_2 = \sum_{u=1}^{N} y_{2u}/N$$

From the correlation analysis it is known that the correlation-coefficient value lies between -1 and +1. If an increase in the value of one response causes the other one to rise, their correlation coefficient has a positive value. The closer a correlation coefficient value is to one, the more the value of one response depends on the value of the other one, i.e. there is a linear connection between responses so that only one response may be followed on the actual research subject. It should be noted once

again that the correlation coefficient has a clear meaning only in the case of the linear relationship and normal distribution of the parameters.

At a high correlation coefficient value, either of the two analyzed responses may be discarded as it adds no new information on the subject of research. Our suggestion is to eliminate the response that is either hard to measure or its physical interpretation is difficult.

Design of experiments insists on measuring all responses and then, by means of correlation analysis, research subject models for the least possible number of responses or for *general response* are made up. This does not mean that there are no cases in practice when correlated responses are used.

Summary

Problems of choosing responses of complex research subjects have been analyzed. The optimization parameter is, in fact, a reaction or response to factor level changes that define the status of a research subject. Responses may be economic, technoeconomic, technical-technological, statistical, psychological, etc. A response should be quantitative, singular, statistically effective, universal, physically real, simple and easily measurable. For responses with no quantitative measurement, the ranking method is used. Out of all responses typical for a research subject, only one or a general response is taken. Other responses are used as constraints.

2.1.2.2 General Response

It is difficult to single out one response as the most important one out of a large number of responses that characterize a research subject. When this happens we have the situation described in the previous chapter. A harder problem is to make up one, a so-called general response [1].

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Each response has its physical sense and its dimension. To join such models, it is first necessary to introduce a non dimensional scale for each response. The scale must be of the same kind for all responses that are generalized. The choice of the scale is not a routine job and it depends on preliminary information we have about the responses and on the precision which is required from the general response.

After choosing the non dimensional scale for each response, one should define the rules of combining *partial responses*. A unique rule or algorithm does not exist.

Simple general response

Assume that a research subject is characterized by n partial responses y_u(u=1, 2,..., n) and that each of these responses is measured in N trials. Then the value of the u-responses in the i-th run is y_{ui} (i=1, 2,..., N). Each of the given responses y_u has its physical interpretation and its dimension. If we introduce the non dimensional scale with only two values 0 and 1, the 0 would correspond to all those values of partial responses that are unsatisfactory by their quality, and the 1 would correspond exactly to those that are satisfactory. The transformed values of partial responses according to the non dimensional scale are marked $\dot{y}_{ui}$. $\dot{y}_{ui}$ and it is the transformed value of the u response in the i-th trial. After the transformation we obtained non-dimensional partial responses that should now be generalized. Since partial responses take

the values 0 and 1, it would be logical to make up the general response with the same values 0 and 1. Thereby the general response should have the value 1 only when all partial responses have the value 1. When only one of the partial responses takes the value 0, the general response must also have the same value. For such imposed conditions the general response satisfies this mathematical expression:

$$Y_i = \left[\prod_{u=1}^{n} \overset{\bullet}{y}_{ui}\right]^{1/n} \tag{2.9}$$

where:
Y_i is the general response in the i-th trial.

$\prod_{u=1}^{n}$ is the multiply of transformed partial responses $\overset{\bullet}{y}_{1i}, \overset{\bullet}{y}_{2i}, \ldots, \overset{\bullet}{y}_{ui}$.

The general response definition by the formula (2.9) may be simplified by deleting the exponent 1/n without affecting its core.

$$Y_i = \prod_{u=1}^{n} \overset{\bullet}{y}_{ui} \tag{2.10}$$

Example 2.1
In developing an optimal technological procedure of producing a new plastic material, the product quality had these seven characteristic responses: y_1 thermostability, y_2 material shining, y_3 keeping of properties at low temperatures, y_4 elasticity module at 20 °C, y_5 tensile strength, y_6 elongation at break and y_7 number of folds before rupture.

These transformations are introduced for the given partial responses.

$$\overset{\bullet}{y}_{1i} = \begin{cases} 1, \text{ if } y_{1i} \succ 100; \\ 0, \text{ if } y_{1i} \leq 100; \end{cases} \quad \overset{\bullet}{y}_{2i} = \begin{cases} 1, \text{ if } y_{2i} \prec 20; \\ 0, \text{ if } y_{2i} \geq 20; \end{cases}$$

$$\overset{\bullet}{y}_{3i} = \begin{cases} 1, \text{ if } y_{3i} \prec -18; \\ 0, \text{ if } y_{3i} \geq -18; \end{cases} \quad \overset{\bullet}{y}_{4i} = \begin{cases} 1, \text{ if } y_{4i} \prec 120; \\ 0, \text{ if } y_{4i} \geq 120; \end{cases}$$

$$\overset{\bullet}{y}_{5i} = \begin{cases} 1, \text{ if } y_{5i} \succ 200; \\ 0, \text{ if } y_{5i} \leq 200; \end{cases} \quad \overset{\bullet}{y}_{6i} = \begin{cases} 1, \text{ if } y_{6i} \succ 200; \\ 0, \text{ if } y_{6i} \leq 200; \end{cases} \quad \overset{\bullet}{y}_{7i} = \begin{cases} 1, \text{ if } y_{7i} \succ 25; \\ 0, \text{ if } y_{7i} \leq 25; \end{cases}$$

Experimental data of the nine trials are given in Table 2.5.

Two general responses are defined for a complex characterization of the material; the first, a general response

$$Y_1 = \left[\overset{\bullet}{y}_1 \times \overset{\bullet}{y}_2 \times \ldots \times \overset{\bullet}{y}_7\right]^{1/7}$$

takes into account the producer's and buyer's demands, while the other general response that considers only the buyer's demands has the form

$$Y_2 = \left[\overset{\bullet}{y}_3 \times \overset{\bullet}{y}_5 \times \overset{\bullet}{y}_7\right]^{1/3}$$

Table 2.5 Original, transformed and general responses

No. trials	Natural, original responses							Transformed, partial responses							General responses	
	y_1	y_2	y_3	y_4	y_5	y_6	y_7	$\dot{y}_1$	$\dot{y}_2$	$\dot{y}_3$	$\dot{y}_4$	$\dot{y}_5$	$\dot{y}_6$	$\dot{y}_7$	Y_1	Y_2
1	272	14	-25	103	215	299	103	1	1	1	1	1	1	1	1	1
2	187	20	–23	91	179	254	29	1	0	1	1	0	1	1	0	0
3	162	21	–24	102	216	270	99	1	0	1	1	1	1	1	0	1
4	461	14	–25	114	198	251	54	1	1	1	1	0	1	1	0	0
5	267	14	–21	105	208	268	31	1	1	1	1	1	1	1	1	1
6	250	24	–27	99	220	304	46	1	0	1	1	1	1	1	0	1
7	489	12	–25	123	201	238	33	1	1	1	0	1	1	1	0	1
8	380	14	–23	116	230	292	126	1	1	1	1	1	1	1	1	1
9	580	29	–22	100	215	304	48	1	0	1	1	1	1	1	0	1

Only three technological procedures may be recommended by the first, general response. If only the producer's demands are considered, the materials obtained in seven trials have a satisfactory quality. If for each of the partial responses we known the best-ideal value to be reached, then the general criterion may be made up by taking into account the given property. Marked with y_{u0} is the best ideal value of response u. We can then consider the difference y_{ui}-y_{u0} as the measure for reaching the ideal value of partial response. The given difference may not be used to define a general criterion for two reasons. The first is that the analyzed difference has the dimension of the associated partial response. The other is that it may have a negative and a positive sign. To switch to a non dimensional value, it is sufficient to divide the observed difference by the associated best value: $(y_{ui}\text{-}y_{u0})/y_{u0}$.

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To eliminate the sign, it is sufficient to square it. In that case the general response is:

$$Y_1 = \sum_{u=1}^{n} \left[\frac{y_{ui} - y_{u0}}{y_{u0}} \right]^2 \tag{2.11}$$

If, in a trial, all partial responses correspond by their values to the associated ideal values, then the general response has the value zero Y=0. That is the general response value that one should try to reach in this case. The closer to zero the better. The deficiency of this procedure for generalization is that each partial response in general response has the same part or the same importance. The practice tells us that all responses are not of the same importance but, moreover, are very different.

The mentioned deficiency may be removed by introducing a *significance coefficient* a_u.

$$Y_1 = \sum_{u=1}^{n} a_u \left[\frac{y_{ui} - y_{u0}}{y_{u0}} \right]^2 \tag{2.12}$$

so that:

$$\sum_{u=1}^{n} a_u = 1; a_u \succ 0.$$

To assert the response significance degree and to determine the significance coefficient, use the method of *expert estimate* [2, 3]. The analyzed algorithms for construing general responses have nevertheless been simple. For more complex general responses it is necessary to define the transformation scale, which will take into account finer differences between partial responses.

The desirability function

The most frequently used general response is *Harington's* [1, 4] *overall desirability function*. The basis of this construction of a general response is transformation of partial responses into a non-dimensional *desirability scale*. To construct a desirability scale we use the prepared, elaborated table of *standard estimates*, Table 2.6.

Table 2.6 Standard estimates on desirability scale

Standard estimates	Desires	Quality of product
1.00	Excellent	The ultimate in "satisfaction" or quality, and improvement beyond this point would have no appreciable value
1.00–0.80	Very good	Acceptable and excellent, represent unusual quality, or performance, well beyond anything commercially available
0.80–0.63	Good	Acceptable and good. represents an improvement over the best commercial quality, the latter having the value of 0.63
0.63–0.37	Satisfactory	Acceptable but poor. quality is acceptable to the specification limits, but improvement is desired
0.37–0.20	Bad	Unacceptable. materials of this quality would lead to failure of the project
0.20–0.00	Very bad	Completely unacceptable

Partial responses transformed into the non dimensional scale are marked d_u(u=1.2,...,n) and called *partial desirability or individual desirability*. As shown in Table 2.6 the desirability scale has the range from 0.0 to 1.0. Two characteristic limit values for quality are within this range 0.37 and 0.63. The 0.37 value is approximately l/e=0.36788, where e is the basis of the natural logarithm, and 0.63 is 1-1/e.

Due to mathematical interpretation of the desirability function, it is rational, convenient and practical to join the desired value d=0.37 to any of the quality properties in a product specification, under the assumption that limit values for the quality really exist. The other practical value of the desirability function or the scale is the limit value 0.63, i.e. the value that corresponds to the best commercial quality of the product, which exists and is acceptable. The mentioned two limit values are geometrically two points of the curve, which is described by the equation.

$$d=\exp[-\exp(-y)]=e^{-e^{-y}} \tag{2.13}$$

The geometric presentation of Eq. (2.13) is in Fig. 2.4. The desirability scale values are inserted on the ordinate from 0 to 1. The response values of the coded dimension (y′) are on the abscissa. The beginning of the abscissa or its null is the exact point to which the ordinate 0.37 corresponds. It should be noted that the point with

coordinates (0; 0.37) corresponds to the first fold point of the curve. The same may be said for the value 0.63. The chosen curve in Fig. 2.4 is adequate to the real situations as it is continuous, monotonous, smooth and besides, the curve ends are less sensitive than the center zone. The coded response or axis y′ is, in principle, divided into 3 or 6 ranges with reference to zero. The choice of number of intervals is important as it determines the curve slope.

Example 2.2

Let one of the responses be a chemical reaction yield with limit values 0.0% and 100.0%.

Assume that the 100% yield is equal to the desirability scale of value 1, and 0.0% to the value 0.

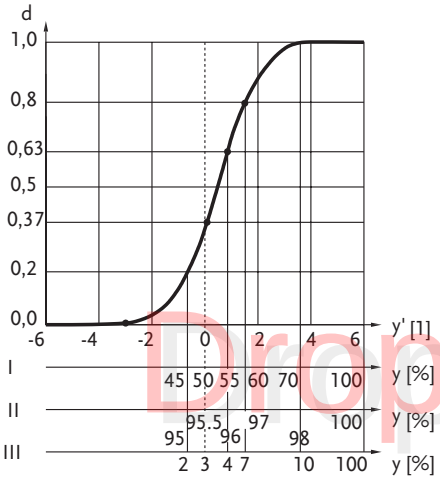

Figure 2.4 Desirability function

The choice of other critical points depends on a series of circumstances such as result requirements, researcher abilities, etc. Take the case where the yield is 50%. The 70% yield is hard to imagine as it may be impossible due to side chemical reactions. After such an assertion it is clear to a researcher that even the 70% yield is the same as that of 100%. That, in fact, is the second point of the researcher's choice and its value on the desirability scale is close to one. The third point should limit the "very good" region, which on the desirability scale is between 0.8 and 1.0. To choose the corresponding response value for this point has so far been the hardest job. If it is hard to obtain a 70% yield, then 60% would definitely be satisfactory, and that is the third point on the abscissa. One should not be sure in this conclusion if the experimental equipment for measuring the yield has a great error and it is unable to differentiate the 60% and 70%. A researcher who is a greater optimist should choose the yield 67% for the required value. For the region of good results (0.80–0.63) he may choose the yield values between 60–55%. The already reached value in the

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experiment of 50% will be taken as the lower limit of satisfactory results. The 45% yield is simply a bad result. The performed correspondence has been geometrically shown in Fig. 2.4 with I as the abscissa. In a case when we dispose with a technological process that has a 95% yield, all this looks different. In that case, greater purity of the product may be demanded and it, apart from other measures taken, may demand a larger yield of 98%. Such a case has been geometrically shown in the same figure by the abscissa II. It should be added that such a desirability scale is possible only through precise yield measurements. The situation is quite different when synthesis of a new product is in question, which so far has not succeeded in giving a new product, even for identification. At a yield of 2%, for example, we are unable even to identify the product and a 10% yield would be a real success. This case is also depicted in Fig. 2.4, as abscissa III.

A curve of desire is often used as a *monogram*. Thus, in the case of I if the yield is 63% one obtains a 0.9 desirability estimate in Fig. 2.4. This procedure of reading the desirability scale from a diagram is often used in practice. In case this method is not precise enough, one uses the analytical method. This means that the coded response y′ is read and then the obtained value is replaced in Eq. (2.13), wherefrom the desirability estimate is calculated. In the previous example only one, quantitative response has been analyzed, it being the chemical reaction yield. The problem gets harder if the qualitative response is in question. In both the first and the second case, it is crucial to determine the acceptable and unacceptable quality limits. Hereby one has to remember that limitations may be *one-sided,* $y_u \leq y_{max}$ or $y_u \leq y_{min}$, and *double-sided,* $y_{min} \leq y_u \leq y_{max}$. Two situations are possible. The first, a simpler one, is when the researcher disposes with information on requirements for each partial response or has clear specifications in which either one or both limitations are defined. Then the estimate on the desirability scale d=0.37 corresponds to y_{min} if we have a one-sided limitation or y_{max} for $y_u \leq y_{max}$. In the case of a double-sided value limitation d=0.37 both y_{min} and y_{max} correspond. In the other situation, the researcher has no specifications available so that the limit values on the desirability scale are determined based on the runs done and the researcher's intuition. It is obvious that in such cases one should not be satisfied with the researcher's opinion and intuition for it can be highly subjective. Therefore opinions of several researchers are used with a check of the degree of accord in their opinions by the *rank correlation* method.

Transformation of partial responses into partial/individual desirability functions

Assume we have an experiment where we dispose with specifications with one or two limit values for each partial response. For those values outside the limit values we have d_u=0, and within them d_u=1. If y_{min} is the lower limit value of the specification and if $y_u \geq y_{min}$ then the *partial desirability function* for a one-sided limitation is:

$$d_u = \begin{cases} 0, \text{ if } y_u \prec y_{\min}; \\ 1, \text{ if } y_u \geq y_{\min}; \end{cases} \tag{2.14}$$

By analogy for a double-sided limitation it is:

$$d_u = \begin{cases} 0, \text{ if } y_u \prec y_{\min} \text{ and } y_u \succ y_{\max}; \\ 1, \text{ if } y_{\min} \leq y_u \leq y_{\max} \quad ; \end{cases} \tag{2.15}$$

In this way we have reached the *simple general response,* which has been analyzed before. The desirability scale has come down to a simple scale with two classes. Both these cases Eqs. (2.14) and (2.15) are shown in Fig. 2.5.

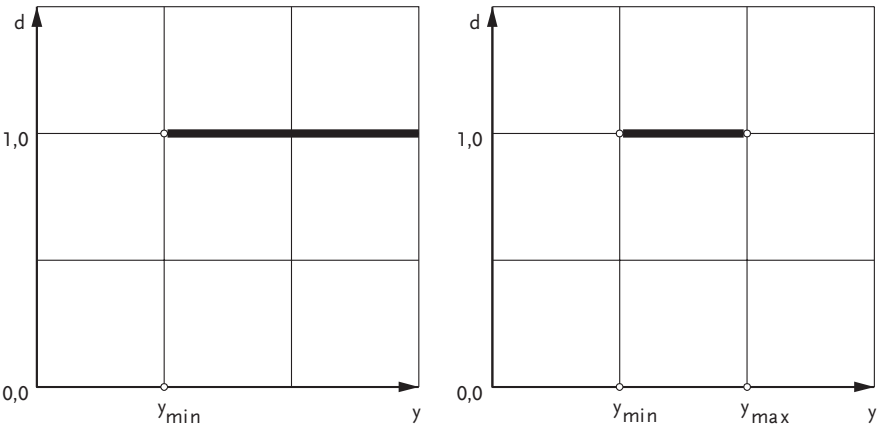

Figure 2.5 Partial desirability function with one- and double-sided limitation

Here we have a very simple classification on acceptable and unacceptable quality, which is rarely met in practice. Transformation of partial responses into a partial desirability, in a large number of cases uses Table 2.6 and desirability (2.13).

For one-sided limitations $y_u \leq y_{max}$ or $y_u \geq y_{min}$, partial desirability, limited on one side, is shown in Fig. 2.6.

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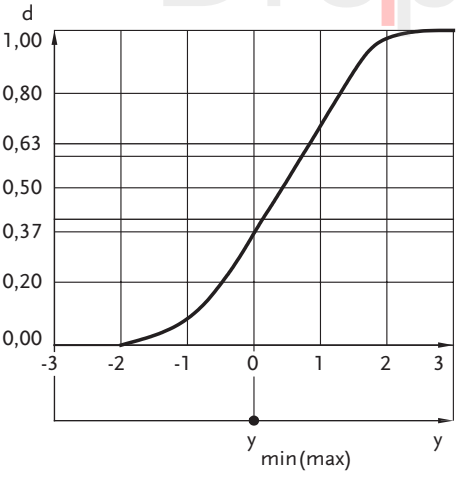

Figure 2.6 One-sided desirability

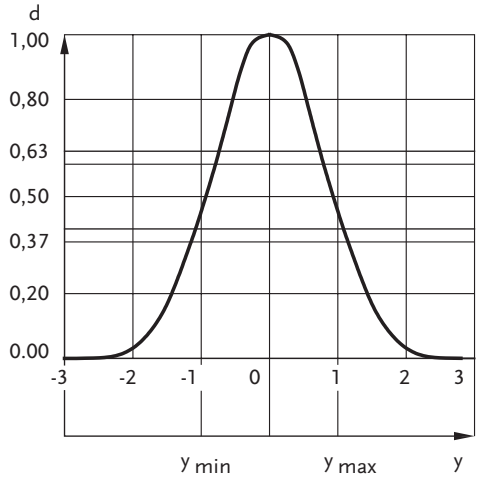

Figure 2.7 Double-sided desirability

Many responses have such one-sided limitations: tensile strength, strain at break, shock toughness, etc. One can see this in Example 2.2 where for all given responses the limitation $y_u \geq y_{min}$ is valid. The other form of limitations $y_u \leq y_{max}$ is typical for responses such as: humidity, specific weight, content of valuable ingredients, etc. Double-sided desirability limitation is shown in Fig. 2.7.

Double-sided limitation $y_{min} \leq y_u \leq y_{max}$ is met more seldom than one-sided and it is more complicated for transformation. The following responses may be mentioned as examples of double-sided limitations: the molecule weight of a material, bulk density, etc. The transformation in Fig. 2.7 is mathematically given as:

$$d = e^{-\left(|y'|\right)^n} \tag{2.16}$$

where:
e is the constant of natural logarithm e=2.71828;
n is the positive number ($0<n<\infty$);
y′ is the linear transformation of the property variable or of partial response y_u ;
y′=–1, when $y_u=y_{min}$ is the lower limit value of specification for the observed quality;
y′=+1, when $y_u=y_{max}$ is the upper limit value of the quality specification;
y′-is the absolute value of y′;

Any value of the partial response (of the observed quality) marked as y_u may be transformed into y′ by means of the expression:

$$y' = \frac{2y_u - (y_{max} + y_{min})}{y_{max} - y_{min}} \tag{2.17}$$

Equation (2.16) is a family of curves for which it is valid that:

- they asymptotically approach d=0 when the absolute value $|y'|$ is above 1.0 ;
- they pass through d=1/e=0.37 when the absolute value $|y'|$ is equal to one $|y'|=1$;

- they pass through d=1.0 halfway between the lower and upper limit values of the product quality specification.

The exponent in Eq. (2.16) determines the curve slope; when n increases the curve approaches faster the limit case d=0.0 outside the specified limits, and d=1.0 between the limit values. For any *desirability curve* that corresponds to Eq. (2.16), n may be calculated by choosing a d value between 0.6 and 0.9, by finding the absolute value y′ and replacing it in the equation:

$$n = \frac{\ln \ln 1/d}{\ln |y'|} \tag{2.18}$$

Overall desirability

After choosing the desirability scale and after the transformation of partial responses into partial desirability it is possible to approach constructing the general response *D*, which is called *Harrington's over all desirability* or *Harrington's general response*. To generalize or switch from d_u to *D* is possible by the formula:

$$D = \left[\prod_{u=1}^{n} d_u \right]^{1/n} \tag{2.19}$$

Equation (2.19) is mathematically the geometric mean of partial responses. The example may be meeting all properties of the material with application requirements. When a property of the material does not satisfy the specification (i.e., the material is brittle and fragile at a certain temperature), then it cannot be used. If a partial desirability is d_u=0, this property must be true of over-all desirability D=0. On the contrary, D=1 when and only when all partial desirability is d_u=1(u=1.2,...,n). Over all desirability is highly sensitive to changes in individual ones. The principle of getting estimates on the desirability scale given in Table 2.6, apart from it being valid for partial ones, is also valid for d_1, d_2, ..., d_n=0.63, D=0.63, or if d_1, d_2, ..., d_n=0.37, D=0.37 too, etc. Over-all desirability includes various partial responses such as: technological, techno economic, physical-chemical, economic, esthetics, etc. Example 2.1 considers construction of an over all response by using the desirability scale with only two values 0 and 1.

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Example 2.3

Based on data on partial responses from Example 2.1, make up Harrington's general response.

As one-sided limited partial responses are in question, use the one-sided limited desirability as given in Fig. 2.8.

Transformed partial responses into partial desirability are shown in Table 2.7. Following *Harrington's* general response:

$$D_1 = \left[d_1 \times d_2 \times d_3 \times d_4 \times d_5 \times d_6 \times d_7 \right]^{1/7}$$

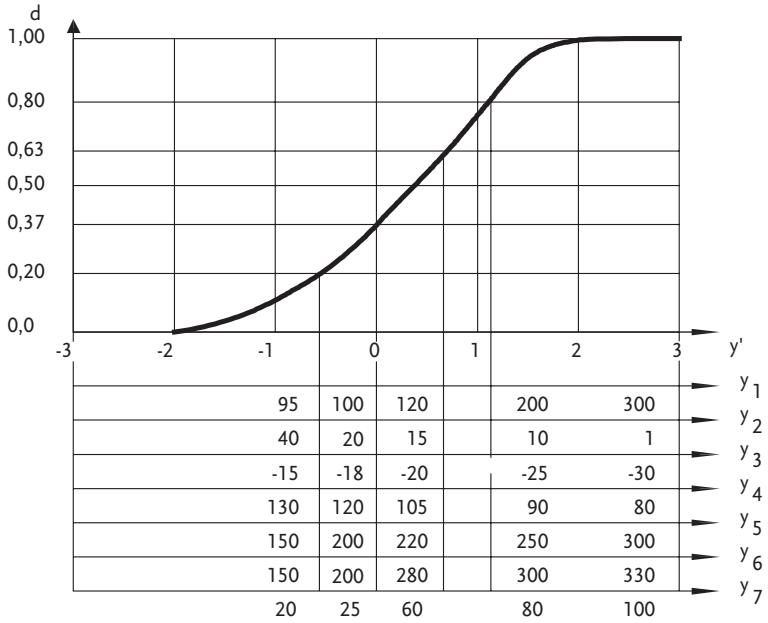

Figure 2.8 Desirability scale

four technological procedures obtained good marks, and five procedures have been satisfactory. By general response:

$$D_2 = \left[d_3 \times d_5 \times d_7\right]^{1/3}$$

which considers only the buyer's demands, three procedures were very good and six satisfactory. By comparing the obtained solutions with those from Example 2.1, it is obvious that *Harrington's* general response is finer.

For obtaining coded values y′ three ranges have been taken in this example or these codes: -3;-2;-1;0;+1;+2;+3. When the desirability curve should be regulated, this may be achieved by changing the number of ranges. To enable the transformation given in Table 2.7, Table 2.6 should be completed by this information:

d_i	y_i'	y_1	y_2	y_3	y_4	y_5	y_6	y_7
1.00–0.80	3.0	300	7	–30	80	300	330	100
0.80–0.63	1.5	200	10	–25	90	250	280	80
0.63–0.37	0.85	120	15	–20	105	220	250	60
0.37–0.20	0.00	100	20	–18	120	200	200	25
0.20–0.00	–0.50	95	40	–15	130	150	150	20

Table 2.7 Partial responses, partial desirability and overall desirability

No. trial	Partial responses							Partial desirability's										
	y_1	y_2	y_3	y_4	y_5	y_6	y_7	d_1	d_2	d_3	d_4	d_5	d_6	d_7	D_1	Mark	D_2	Mark
1	272	14	–25	103	215	299	103	0.98	0.67	0.80	0.71	0.55	0.80	1.00	0.77	G	0.76	G
2	187	20	–23	91	179	254	29	0.77	0.36	0.73	0.79	0.32	0.53	0.29	0.50	S	0.41	S
3	162	21	–24	102	216	270	99	0.68	0.35	0.75	0.72	0.55	0.56	0.99	0.63	G	0.74	G
4	461	14	–25	114	198	251	54	1.00	0.67	0.80	0.47	0.38	0.53	0.57	0.60	S	0.56	S
5	257	14	–21	105	208	268	31	0.97	0.67	0.70	0.63	0.44	0.54	0.39	0.60	S	0.49	S
6	250	24	–27	99	220	304	46	0.95	0.32	0.90	0.70	0.63	0.81	0.48	0.65	G	0.62	S
7	489	12	–25	123	201	238	33	1.00	0.72	0.80	0.35	0.38	0.50	0.40	0.53	S	0.50	S
8	380	14	–23	116	230	292	126	1.00	0.67	0.73	0.44	0.67	0.79	1.00	0.73	G	0.79	G
9	580	29	–22	100	215	304	48	1.00	0.30	0.72	0.70	0.55	0.81	0.52	0.63	S	0.60	S

Remark: $D_1=[d_1 \times d_2 \times d_3 \times d_4 \times d_5 \times d_6 \times d_7]^{1/7}$ $D_2=[d_3 \times d_5 \times d_7]^{1/3}$
G-good mark **S-satisfactory mark**

Overall desirability is an abstract definition and therefore some of its properties have to be analyzed, such as: lack of fit and statistical effectiveness. It has been asserted that the effectiveness and sensitivity of partial and overall desirability are not lower than the same properties of any technological response. Overall desirability is quantitative, singular, statistically effective, adequate, etc. It has found a large application in the research of polymeric materials, rubber products, etc.

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2.1.2.3 Ranking of the Qualitative Responses

Among the response requirements of a research subject that have to be met in the first place is that it has to be quantitative. A researcher usually keeps to this requirement, however there are situations when it cannot be met, and the researcher has to deal with qualitative responses. Due to the fact that in the case of qualitative responses the efficiency of experimental research is reduced, one should try to transform these responses into quantitative ones. For this, one may use the transformation of qualitative response by desirability scale into partial desirability.

Example 2.4 [5]

In a full-scale plant for producing double-base propellants a study was done to discover a high-energetic propellant with a high burning rate and low temperature sensitivity. The problem of making such a propellant consisted in a high percentage of ignitions, even up to 66% of the total number of batches. In the discovery and the elimination of inflammability causes of certain batches when gelled on rollers, a great problem was the qualitative response of propellant inflammability, or lack of possibility to quantitatively express the propellant ignition at gelling. The research program for discovering these causes included eight trials that were repeated once. It should be noted that as a correct propellant is production in this case is considered, the propellant produced after 30 passes over rollers for gelling. The data of all trials are shown in Table 2.8. Do the ranking of the qualitative response.

Table 2.8 Ranking of the qualitative response

No. of trials	I Production-qualitative response	Rank	II Production-qualitative response	Rank	$\bar{d}_n$
1	Done with no problems	1.00	Done with no problems	1.00	1.000
2	Done with crackling	0.85	Done with no problems	1.00	0.925
3	Ignition in 22 passes	0.58	Ignition in 10 passes	0.37	0.475
4	Ignition in 17 passes	0.44	Done with no problems	1.00	0.720
5	Done with no problems	1.00	Done with crackling	0.78	0.890
6	Ignition in 25 passes	0.68	Ignition in 20 passes	0.53	0.603
7	Ignition in 15 passes	0.54	Done with no problems	1.00	0.770
8	Done with crackling	0.92	Done with no problems	1.00	0.960

Ranking is done by using the one-sided desirability given in Fig. 2.9.

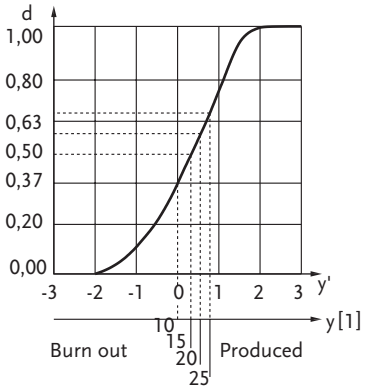

Figure 2.9 Response ranking by desirability scale

Summary

The construction of the general response is linked to defining one quantitative response to a research subject with several partial responses, each of which has its own physical interpretation and dimension. To form from such different partial responses a unique response, it is necessary to transform all partial responses into non dimensional values by a unique scale. It is therefore necessary when defining a general response first to choose the scale for doing the transformation. The scale must be unique for all partial responses to be transformed. The choice of the scale depends on preliminary information about partial responses and on the required precision of the general response.

The next problem is choosing the rule by which the transformed partial responses will be combined into general response. There is no rule, and the way to choose the combinations is not defined. Certain approaches that a researcher might use have been presented.

2.1.3 Selection of Factors, Levels and Basic Level

Having selected the system response, we start choosing factors, levels of the factors and *center point of the design (basic level* or the *null point)*. By factor we understand the controllable independent variable that corresponds to one possibility of influence on the object of research. A factor is considered defined if its *name* and *domain of factors* are determined. A factor may take several values in this field. The chosen factor values, both qualitative and quantitative, are called factor *variation levels*. Factor variation levels in the design of experiments are coded values. Under *factor interval of variation* we understand the difference between two factor levels, which in their coded form have value one. When selecting the factors one should pay attention to the conditions they must meet.

Factors should be:

- controllable
- of high measurement precision
- singular
- concordant
- noncorrelated linear-wise.

The controllable requirement of factors is linked to the possibility of setting them on several levels and maintaining those levels precise enough. Or, by changing factor values, one changes the research subject status or controls the subject.

Factor singularity means its property to directly change the status of a research subject, i.e. it is not a function of other factors and it may be fixed to any value in the domain of factors.

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Factor concordance is a property that makes it possible for all factor combinations to be realized in an experiment. This property is very important when an experiment with several simultaneous factor variations is designed. It is not a rare case where the lack of this property brings about a change in defining a research problem, excluding some factors from the experiment, or it changes the domain of factors.

The question of linear correlation between factors deserves special attention. There is a rule saying that in the case of a linear correlation between factors it is impossible to design an experiment. This is connected with the requirement to keep in each *design point of experiment-trial* (one combination of factor levels) each factor at a corresponding level, independent from the others. Besides, in the case of a linear correlation between two factors, it is sufficient for only one of them to be included in the experiment, for inclusion of the other one does not offer any additional information on the research subject. The optimization problem is often complicated if all the observed factors can not be expressed quantitatively. The existence of categorical/qualitative factors is connected with insufficient knowledge of the researched phenomenon or subject of research. Through a better level of knowledge about the research subject, categorical/qualitative factors change into quantitative ones. When categorical/qualitative factors are present, an optimization problem may be solved in two ways:

- separately for each level of categorical/qualitative factor and then by comparing the obtained optimal solutions;
- simultaneously for all levels by quantitative defining of a factor on several levels.

Selection of one of these two ways depends on the particular problem. The first one mostly gives more reliable results but requires a longer time and is more costly. When we have good preliminary information on a research subject, we may use, in an experiment, the *complex factors*-similarity criteria, component concentration, logarithms, simplexes of geometric dimensions, etc. [6]. When defining factors it is important to take all those potential factors that may affect the research subject. If we forget one of the crucial factors, this eventually may have very bad consequences for the researcher. Namely, a forgotten factor will, during the experiment, act randomly taking random values out of the researcher's control, which means that the value of a *trial error* will increase. When the forgotten factor remains at a fixed level we may infer a false optimum as there is no guarantee that the fixed level of the factor is optimal.

In practice we are often faced with a research subject that has several technological phases and where the response is measured in its last phase. In that case, the subject is studied cybernetically as a *"black box"*, like a unique technological phase with all the factors that corresponded to individual technological phases. We had no responses by individual technological phases in this case, but this may occur. Moreover, response optima by individual phases contradict the general optimum system. This indicates that optimization by individual phases of a research subject is justified and possible. In this way it is possible to incorporate into the design of an experiment, factors from various phases of a research subject, but this is not always necessary.

When selecting a domain of factors one should pay special attention to choosing the *center point of the design (basic level* or the *null point)*. The choice of a null point is associated with selection of the initial status of the research subject to perform optimization. As optimization is connected with improvement of the subject status in comparison with the status in the null point, it is desirable that the point is in the optimum region or as close to it as possible. If the mentioned research was preceded by other experiments on the same subject, the status having the most convenient response value is taken as the null experiment. The null point is quite often the center of the domain of factors. The most important alternatives in selecting the basic and null levels are shown in Fig. 2.10.

Having defined the null point, we choose the *factor intervals of variation*. The selection of these factors means determining such factor values, which in their coded form have the values +1 and -1. When choosing this factor in the *experimental domain* we obtain a subdomain, symmetrical to the null point, which is used in the first experimental phase. When choosing the factor interval of variation one must keep in mind the fact that factor values corresponding to levels +1 and -1 must be different enough from those that correspond to the null level. Therefore in almost all cases, the variation interval (ε) is taken as twice as large as the *error fixing factor*. Too large a factor variation interval is also a problem, for it reduces the efficiency of finding an optimum, especially in regards to the *steepest ascent method*. On the contrary, a small variation interval does not present a problem in practice, since the

domain of factors is generally known in advance, including the information on expected order of the mathematical model. The variation interval must not be too small, for in that case, the response effects may not be registered. Block schemes are shown in Figs. 2.11–2.13.

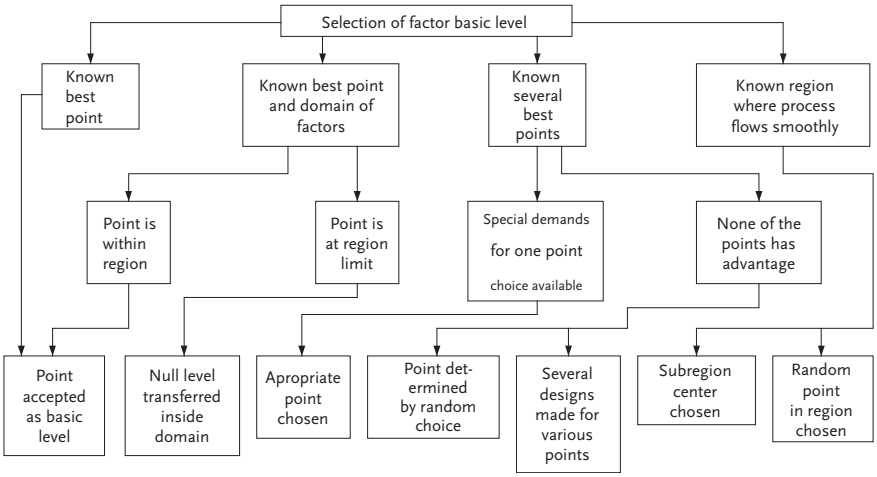
Selection of factor basic level
Known best point
Known best point and domain of factors
Known several best points
Known region where process flows smoothly
Point is within region
Point is at region limit
Special demands for one point
choice available
None of the points has advantage
Point accepted as basic level
Null level transferred inside domain
Apropriate point chosen
Point determined by random choice
Several designs made for various points
Subregion center chosen
Random point in region chosen

Figure 2.10 Block diagram for choice of center point

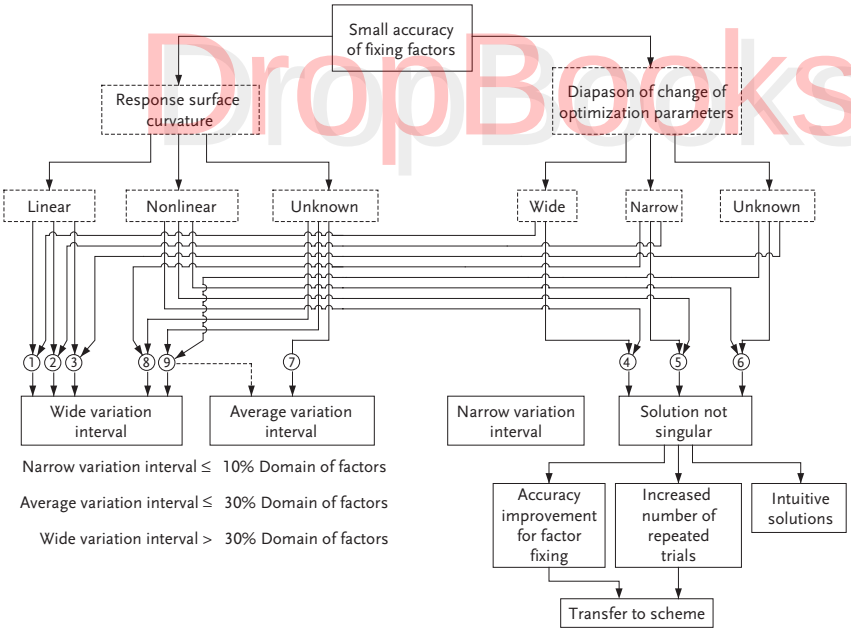

Figure 2.11 Block diagram of accepting factor variation intervals

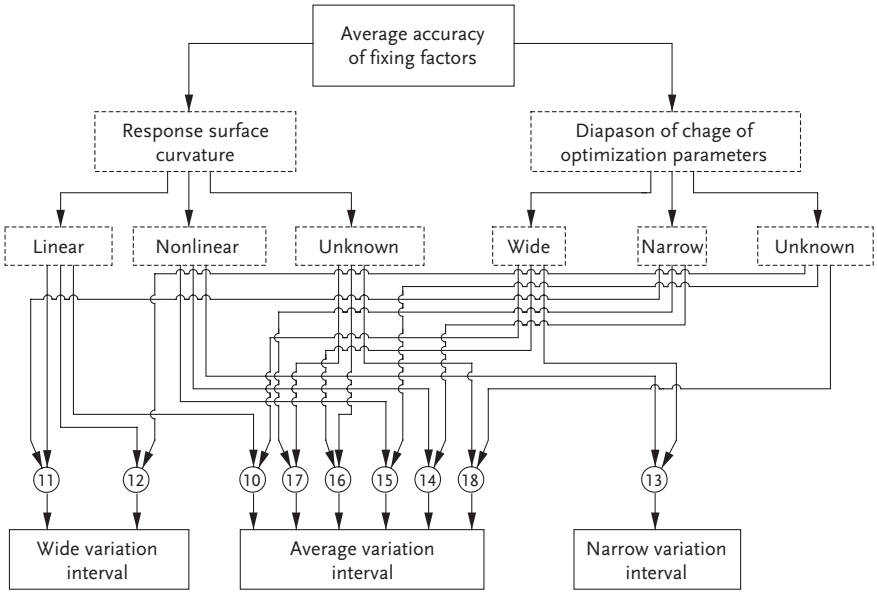

Figure 2.12 Block diagram of accepting factor variation intervals

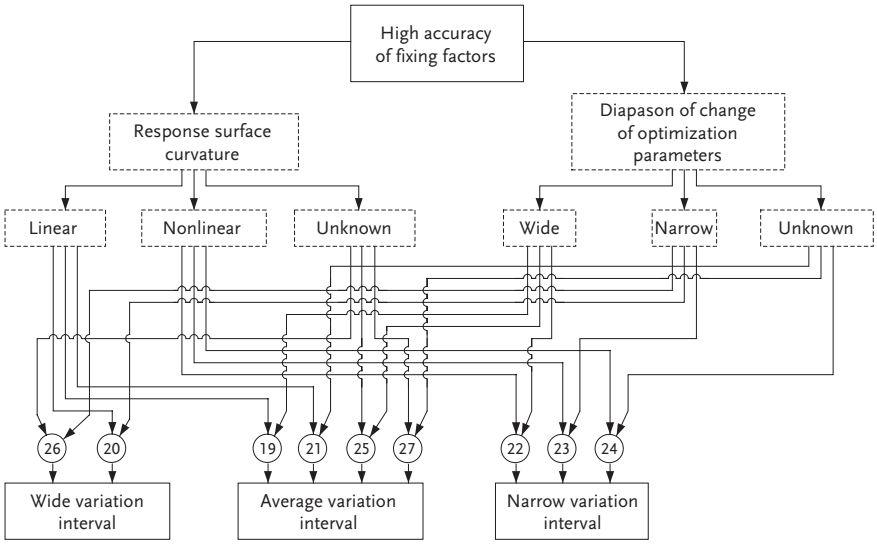

Figure 2.13 Block diagram of accepting factor variation intervals

The presented block diagrams link the factor-fixing accuracy, range of response change and response-surface curvature with the width of factor-variation interval. When selecting a factor variation interval one should, if possible, account for the number of factor variation levels in the experimental domain. Depending on the number of these levels, are the experiment range and optimization efficiency.

In a general case, a design point number (number of trials – different stages of research subject) depends on factor level number and is written:

$$N=p^k \tag{2.20}$$

where:
N is number of design points – trials;
p is number of factor levels and
k is number of factors.

The relation (2.20) is correct for the case of the same number of variation levels of each factor. The minimal number of factor level variations is two and it is most frequent in the first phase of research. Those are *upper* and *lower* levels marked as +1 and -1. Factor variations on two levels are applied in *screening experiments*, in the phase of movement to the optimum and when describing the research subject by linear models. This number of factor levels is not sufficient to obtain second-order models, for a set of lines of different degrees of curving may be drawn through the two points. With an increased number of factor levels, experimental sensitivity is raised, but also the number of design points. To obtain a second order model it is necessary to do an experiment where factors vary at three, four or more levels. In our case, the number of factor variation levels is determined in accord with the research conditions and the plotted design of experiment. Hence problems may appear when the research includes categorical/qualitative factors or those that change discretely. A categorical/qualitative factor, for example, has no evident physical sense for the null level. This deficiency of categorical/qualitative factors does not affect optimization efficiency in the case of the linear model. The situation is more complicated when, in modeling the second order, one must account for categorical/qualitative factors (a factor must be varied at least at three levels). Accounting for these deficiencies, it is recommended to include categorical/qualitative factors only in the screening experiments and in the methods of designing experiments, which have nothing to do with obtaining nonlinear models, such as: analysis of variance, random balance method, full-factorial designs on two levels, etc. Factor selection is completed by making a list of all factors that are of interest in the researcher's opinion. Thereby, factor names and marks, their ranges, variation levels and null-point coordinates, are defined.

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It is important once again to note that, when considering factors, all variables having the least possible chance to affect the research subject are included. It is better in such a situation to include more factors, for the nonessential ones will be rejected in the process of selection. An example of defining factors is shown in Table 2.9:

Table 2.9 Selections of factors

Name and mark of factor	Variation levels					Variation interval ε
	–2	–1	0	+1	+2	
Temperature, °C-X_1	140	150	160	170	180	10
Pressure, bar-X_2	0	2.5	5	7.5	10	2.5
Concentration, g/cm^3-X_3	0	10	20	30	40	10
Time, min-X_4	30	60	90	120	150	30
Mass, kg-X_5	100	160	200	250	300	50

From the total number of noted factors, the researcher chooses those that may be varied during the experiment while he keeps the others at constant levels. When the number of selected factors is more than seven, a possibility of doing the screening experiments has to be considered. For a relatively small number of factors, an experiment is done to reach the optimum or to obtain the mathematical model of the research subject, depending on what the objective of the researcher's problem is.

Finally, we call the extreme values those factors can take, without changing the physical-chemical properties of research subject, *physical limits of factors*, and the interval X_{1max}-X_{1min} *domain of factors*. Geometric interpretation is shown in Fig. 2.14.

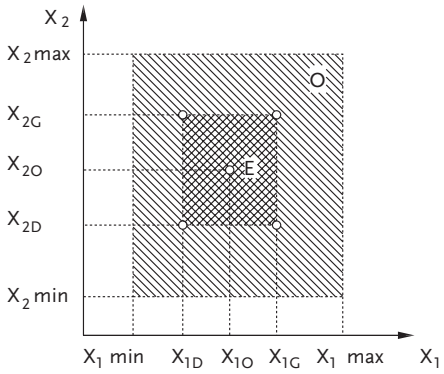

Figure 2.14 Domain of factors

Domain of factors is marked *"O"*. The figure clearly shows that intervals of factor variations are part of the domain of factors when the optimization problem is being solved. This is necessary in order to realize movement towards optimum in this domain. The experiment domain is in the same figure marked by letter *"E"*. In studies with an objective of approximation or interpolation, that is mathematical modeling, the factor-variation intervals cover the whole of the domain of factors. For a two-factor experiment the upper level of factors X_1 and X_2 corresponds to values X_{1max},- and X_{2max}, while the lower levels have values X_{1min}, X_{2min}. Domain of factors *"O"* is in that case called *interpolational*, and *"E"* the *domain of extreme experiment*.

Summary

In this section we have defined a factor as a variable that may affect the research subject. For a variable to be a factor, it must, besides others, fulfill the requirement that it is controllable and singular.

To control a factor means to bring it to a corresponding value or level and keep it constant during a design point-trial, or to change it by a previously set up program. This is exactly the special property of an active or designed experiment. Design of an experiment is possible only in a case when a researcher may, according to his own program, assign the associated values or levels to factors.

Factors should directly change the research subject state. It is hard to control a factor that is a function of other variables, but this does not mean that in a design of experiments, complex factors, such as logarithms, similarity criteria etc., may not be used. Besides the mentioned requirements, factors should be concordant and linearly uncorrelated. When some of the significant factors have been left out in selection, a researcher may get a wrong optimum or a big trial error. Factors can be qualitative/categorical and quantitative. The accuracy of fixing a factor should be high and should depend on the factor variation range. Selection of a factor is especially important in defining a research problem and the result of experimental research greatly depends on it.

2.1.4 Measuring Errors of Factors and Responses

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An important property of design of experiments is a search for increased accuracy in fixing a factor and measuring an error. The researcher must be able to determine and estimate a measurement error correctly. Measurements and measurement errors are a subject of special study, see [7, 8].

Measurement should not be brought down to simply determining a measured value but also to estimating errors in measurements, called the *measurement error*. There are several kinds of errors in measurement: *robust, systematic* and *random*.

Robust errors result from disrupting basic conditions for measuring, researcher's error, etc. A researcher is asked to check the probability of appearance of a robust error. A robust error appears as a measured value that is drastically different from others. This error may be avoided if another researcher who is ignorant of former measurements repeats it. The same effect may be achieved when the same researcher repeats measurements after some time when he has already forgotten the results the of first ones. Such a result has to be rejected if a robust error has been discovered.

Systematic errors appear as a result of the activity of certain factors and in cases of numerous repetitions of the same measurement. This kind of error occurs when measuring is done with an instrument with incorrect calibration. A systematic error is discovered by measurements with different instruments or different methods of the same magnitude. We distinguish among several kinds of systematic errors: known nature and unknown magnitude and systematic errors of unknown origin. Systematic errors of known origin and magnitude are not a problem as they may be

included into measurement results as corrections. The problem is the other aspects of systematic errors, *error theory*, which is based on theoretical probability laws. As processing the results of designed experiment accepts only random errors, only this kind of error is the subject of analysis. Random measurement errors are characterized by an associated distribution law. The distribution of random errors is mostly suited to the normal distribution law given in Sect. 1.1.3. Normal distribution is defined by the arithmetic mean of random value $\bar{X}$ and sample variance S^2. The value $\bar{X}$ is the most probable value of measured property and is calculated by the well-known formula:

$$\bar{X} = \sum_{i=1}^{u} X_i / u \tag{2.21}$$

where:
X_i are measured values;
u is number of repeated measurements.

Variance value or variance measurement is in this case also determined by the well-known formula:

$$\sigma^2 \approx S^2 = \frac{\sum_{1}^{u} \left(X_i - \bar{X}\right)^2}{u-1} \tag{2.22}$$

The positive value from the variance measurement root square is called the *error mean square* or *standard error.*

$$\sigma \approx S = +\sqrt{S^2} \tag{2.23}$$

When estimating measurement results, the important thing is not only to know its accuracy but also the *measurement confidence*. The degree of measurement the confidence is estimated from confidence interval as defined by the level of significance. Let X denote the actual measurement value and ΔX the error in measuring the mean $\bar{X}$, then:

$$P\left(\bar{X} - \Delta X \leq X \leq \bar{X} + \Delta X\right) 1 - \alpha \tag{2.24}$$

where 1-α is the confidence coefficient or the probability that the measurement result is within the confidence interval (2.24). For a 5% level of significance, the confidence interval limits for the measurement mean may by determined if we know the measurement variance for a corresponding number of measurements:

$$X = \bar{X} \pm 1.96 \frac{S}{\sqrt{u}} \tag{2.25}$$

This indicates that to know the measurement random error it is not sufficient to know its magnitude only (confidence interval of measurement error) but also the significance level that facilitates the confidence estimate of the obtained measurements. Using the error mean square as a measurement accuracy property is convenient because that value in a normal distribution is associated with a confidence or confidence coefficient of 0.68 probability. The doubled error mean square 2S has

0.95 confidence and 3S has 0.997 confidence level. To know the error mean square indicates a possibility to establish the measurement confidence interval for any confidence coefficient. Table 2.10 is suitable for such calculations, which for the associated confidence 1-α contains ΔX values expressed as error mean square (θ=ΔX/S),

Table 2.10 Confidence interval coefficient

θ=ΔX/S	3.9	2.6	2.4	2.0	1.65	0.7	0.3	0.15	0.05
1-α	0.9999	0.990	0.984	0.950	0.90	0.51	0.24	0.12	0.04

Example 2.5

100 measurements (u=100) were done for an unknown property. Using expression (2.21) the mean of all measurements $\bar{X}$=1.27 was determined. The calculation by expression (2.22) offered the measurement error mean square S=0.032. Determine the measurement confidence interval for confidence coefficient 1-α=0.98.

Table 2.10 gives for the associated confidence that θ=2.4, so that:

$\Delta X=\theta\times S=2.4\times 0.032=0.08$

The measurement confidence interval is:

$$\bar{X}-0.08\le X\le \bar{X}+0.08$$

$1.19\le X\le 1.35$ or $X=1.27\pm 0.08$

This problem may set up another problem. What is the probability for $\bar{X}$=1.27 and S=0.032 results of individual measurements not to fall outside the measurement confidence interval $1.19\le X\le 1.35$?

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For ΔX=0.08 we obtain:

$$\theta=\frac{\Delta X}{S}=\frac{0.08}{0.032}=2.4$$

Probability 0.984 corresponds to the value θ=2.4 in Table 2.10. Hence 98.4% of all individual measurements fall within their confidence interval. The confidence interval of individual measurements has evidently been analyzed so far. However, in practice it is very important to know the deviation of the arithmetic measurement mean from the actual X value. This problem was more generally solved in Sect. 1.3.2. Hence, the value ΔX is determined as follows:

$$\Delta X=\pm\frac{t\times S}{\sqrt{u}} \tag{2.26}$$

where:
t is Student's distribution, Table C;
S is error mean square of measurements;
u is number of measurements.

We know that with an increase in confidence or its coefficient the t-value also rises, which means that ΔX also goes up resulting in a decrease of accuracy in determining X. In accord with Eq. (2.26), to maintain accuracy in measuring X, it is nec-

essary to reduce the error mean square of measurement S or to increase the number of measurements u. Eq. (2.26) has this form when we consider the relation:

$$P\left[\bar{X} - \frac{t \times S}{\sqrt{u}} \leq X \leq \bar{X} + \frac{t \times S}{\sqrt{u}}\right] = 1 - \alpha \tag{2.27}$$

Equation (2.27) is used to determine the confidence interval or its limit of arithmetic measurement mean, to the actual measurement value for the given confidence coefficient and the number of measurements.

Example 2.6

Determine confidence interval limits within which is the average measurement value at α=0.05. Five measurements were done (u=5). The arithmetic mean is $\bar{X}$=31.2 and S=0.24. From Table C for α=0.05 and f=u-1=5-1=4 we obtain $t_{0.05}$=2.78 so that:

$$\Delta X = \pm\frac{2.78 \times 0.24}{\sqrt{5}} = \pm 0.30$$

31.20-0.30≤X≤31.20+0.30

X=31.20±0.30

Hence we may assert with 0.95 confidence that the actual measurement value is between 30.90 and 31.50. For the same values from Example 2.6 we may ask the following question: What is the probability or confidence that average measurement value $\bar{X}$=31.20 does not differ from its actual value by more than 0.20? By using Eq. (2.26) we get:

$$t = \frac{\Delta X \times \sqrt{u}}{S} = \frac{0.20 \times \sqrt{5}}{0.24} = 1.86$$

For the obtained arithmetic value of Student's criterion t=1.86 and for f=u-1=4 from Table C we have α=0.14 or 1-α=0.86. Analogous calculations show that for the same error mean square of measurements and for the same ΔX=0.20, an increase in the number of measurements to 10 (u=10) allows an increase in confidence to 0.97, for:

$$t = \frac{0.20 \times \sqrt{10}}{0.24} = 2.6$$

Hence, calculations from relation (2.26) facilitate determining the necessary number of measurements (u). Thereby, it is of course necessary to previously define the size of the random value that may be accepted and the coefficient or degree of measurement confidence. In practice, we are satisfied with the level that is not above 0.5%. Table 2.11 is used for practical determination of the necessary number of measurements, for known measurement confidence 1-α and for different confidence interval limits expressed by the error mean square of measurement ΔX/S.

Table 2.11 Number of measurements

ΔX/S	Necessary number of measurements 1-α			
	0.90	**0.95**	**0.99**	**0.999**
1.0	5	7	11	17
0.5	13	18	31	50
0.4	19	27	46	74
0.3	32	46	78	127
0.2	70	99	171	277

If it comes out that to reduce random error it is necessary to increase the number of measurements drastically, it is more acceptable to try to find a way to reduce random error by increasing measurement accuracy or by reducing the error mean square of measurement S. This may be achieved by changing the measurement method or using more up-to-date equipment. Knowledge of the error mean square of measurement obtained from its results may be used to discover robust (extreme) measurement values. When a researcher thinks that a measurement has an extreme value, then the following Student's t-criterion value is calculated:

$$t = \frac{X_E - \bar{X}}{S} \tag{2.28}$$

where:
X_E is extreme measurement value;
$\bar{X}$ is arithmetic mean of other measurements but without extreme.

The calculated t-criterion value is then compared with the tabular value for the associated degree of freedom and significance level. When the calculated value is above the tabular, it means that the extreme measurement value is a robust error and it should be rejected.

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Example 2.7

The mean $\bar{X}$=6.500 from u=41 was obtained in measuring a property. The associated error mean square has a S=0.133 value. The researcher assumes that singular measurement X_E=6.866 is a robust error.

Checking shows this:

$$t = \frac{6.866 - 6.500}{0.133} = 2.75$$

From Table C we obtain t_T=2.74 for confidence level α=0.01 and f=u-1=41-1=40. Since t_R=2.75>t_T=2.74 it confirms that the researcher was right and that the analyzed measurement should be dropped. Note that the same procedure of rejection of extreme values was demonstrated in Sect. 1.5.

When doing experimental research, one should distinguish several kinds of errors: *measurement error, trial error* and *experiment error*. These errors will be analyzed in detail in a subsequent chapter.

2.2 Screening Experiments

2.2.1 Preliminary Ranking of the Factors

We shall here consider the methods that are applied in processing reference data and which simultaneously serve as the first phase of experimental research in cases when from the total number of factors we should select the most important ones. In this phase of formalizing the preliminary information, it is very useful to apply a *psychological experiment*. This experiment is a method of objective processing of the data obtained from either researchers, specialists in the observed field, or reference literature. This kind of experiment facilitates objective knowledge of a research subject, accepting or rejecting of preliminary stated hypotheses, objective comparison of effects of different factors on system response and, hence, a correct selection of factors for the *active* experiment phase. The method of *preliminary ranking of the factors*, is based on the methods of rank correlation [9]. The subject of this method is that factors, in accord with preliminary information, are ranked according to the order of their effects on the response system. The effect of each factor is judged by the *rank-place*, each researcher has given to it (based on the researcher's enquiry, expert papers, literature, etc.) in ranking all the factors by their assumed effect (quantitative effect unknown) on response. When gathering information from each researcher, he is required to fill in the enquiry on the order of effects of the given factors on a certain response. The enquiry includes factors, their dimensions and assumed variation intervals. The researcher fills in the enquiry by defining the place of each factor in a ranking order. Each enquired researcher may, simultaneously, supplement the enquiry by new facts and suggested variation intervals. The enquiry results or ranking by reference data is processed in this way.

First sums of ranks by factors ($\sum_{1}^{m} a_{ij}$), then differences (Δi) between sums of ranks for each factor and average sums of ranks and sums of squares deviations (S) are determined:

$$\Delta i = \sum_{1}^{m} a_{ij} - \frac{\sum_{1}^{k}\sum_{1}^{m} a_{ij}}{k} = \sum_{1}^{m} a_{ij} - T \tag{2.29}$$

$$S = \sum_{1}^{m} (\Delta i)^2 \tag{2.30}$$

where:
a_{ij} is rank of factor i with researcher j,
m is number of researchers,
k is number of factors,
T is average sum of ranks.

The obtained results facilitate constructing the rank graph. However, prior to that, one should determine the degree of opinion concordance of all the researchers by the *concordance coefficient*:

$$\omega = \frac{12 \times S}{m^2\left(k^3 - k\right) - m\sum_{1}^{m} T_j} \tag{2.31}$$

where:

$$T_j = \sum\left(t_j^3 - t_j\right)$$

t_j is number of equal ranks in ranking j.

Before drawing a conclusion based on the concordance coefficient, it is necessary to test its significance. Thereby one should keep in mind that value $m(k-1)\omega$ has an χ^2 distribution with f=k-1 degrees of freedom. The arithmetic value of the χ^2 criterion is calculated by the formula:

$$\chi^2 = \frac{12 \times S}{mk(k+1) - \frac{1}{k-1}\sum_{1}^{m} T_j} \tag{2.32}$$

The hypothesis on concordance of the researchers' opinions is accepted if by the given degrees of freedom and α significance level the calculated value χ^2 is above the tabular one [10].

Starting with concordance of the researchers' opinions, the rank graph is constructed by inserting factors on the abscissa and the associated sums of ranks on the ordinate, but in the opposite direction. Due to the direction of inserting the sums of ranks, the larger rectangle above the abscissa corresponds to a smaller sum of ranks. Depending on the shape of the *rank curve*, which connects the histogram rectangles, different decisions may be made.

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- When the rank curve has the shape as in Fig. 2.15, factors from the first half of the graph enter the basic experiment;

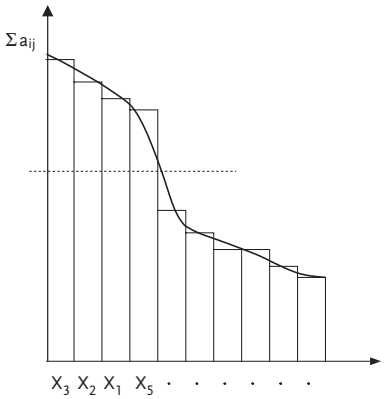

Figure 2.15 Rank graph

- When the rank curve is continually decreasing as in Figs. 2.16 and 2.17, all factors are included into the next active experiment for selecting factors.

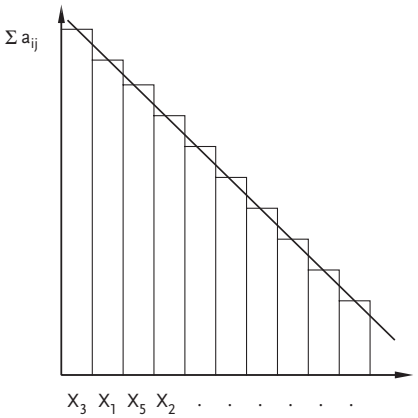

Figure 2.16 Rank graph

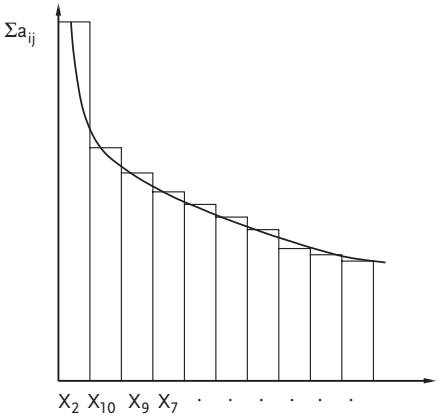

Figure 2.17 Rank graph

Example 2.8 [11]

A composite rocket propellant is mixed up in a vertical planetary mixer. The viscosity of the mixed propellant depends on eight factors according to the reference literature. Those factors are: X_1 mixing temperature; X_2 time of mixing after addition of the third portion of ammonium perchlorate; X_3 mixing rate; X_4 mixture mass; X_5 mixing time after addition of the first portion of ammonium perchlorate; X_6 mixing time after addition of the second portion of ammonium perchlorate; X_7 mixing time of premix and X_8 vacuum in the mixer. The outcomes of the inquiry by eight researchers are presented in Table 2.12. Complete factor selection based on the significance of their effect on dynamic viscosity, by applying the method of prior ranking of the factors.

The following values may be taken from Table 2.12:

$$\sum_{1}^{n} T_j = 120; \quad T = 36; \qquad S = 2019.50; \quad \text{so that:}$$

$$\omega = \frac{12 \times 2019.50}{8^2\left(8^3 - 8\right) - 8 \times 120} = 0.77$$

Since the concordance coefficient values are significantly different from zero, one may say that there is concordance among the opinions of eight researchers. To be sure, the significance of the concordance coefficient was checked by Eq. (2.32).

$$\chi^2 = \frac{12 \times 2019.50}{8 \times 8(8+1) - \frac{1}{8-1} \times 120} = 43.36; f = 8 - 1 = 7.$$

Table 2.12 Results of ranking factors

Researchers m	a_{ij}								$T_j = \sum_{i-1}^{8} \left(t_j^3 - t_j\right)$
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	
1	1	2	6.5	6.5	4	4	4	8	24+6=30
2	1	2	3.5	3.5	7.5	7.5	5.5	5.5	6+6+6=18
3	1	2.5	2.5	5	5	5	7.5	7.5	6+6+6=18
4	2.5	1	2.5	4	5.5	5.5	7	8	6+6=12
5	1	2.5	2.5	4	5.5	7	8	8	6+6=12
6	1	3	2	4	5	6	7	8	0
7	1	2	3	4	5	6	7	8	0
8	1	2.5	4.5	6	2.5	4.5	8	7	0
$\sum_{1}^{m} a_{ij}$	9.5	19.5	26.0	36.0	40.0	44.0	53.0	60.0	$\sum_{1}^{m} T_j = 120$
Δ_i	-26.5	-16.5	-10.0	0.0	4.0	8.0	17.0	24.0	
$(\Delta_i)^2$	702.25	272.25	100.00	0.0	16.0	64.0	289.0	576.0	$\sum_{1}^{k} (\Delta_i)^2 = 2019.50$

From Table D for α=0.05 and degrees of freedom f=7, the tabular value is $\chi^2_{7;95\%}$=14.1. Since the calculated value of, χ^2 criterion is above the tabular value, the hypothesis on concordance of the researchers' opinions is accepted. This conclusion allows us to construct the rank graph in Fig. 2.18. The rank graph clearly shows an equal distribution, so these eight factors should be included in the active experiment for factor selection.

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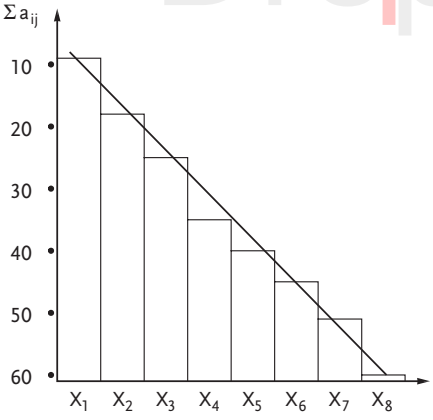

Figure 2.18 Rank graph

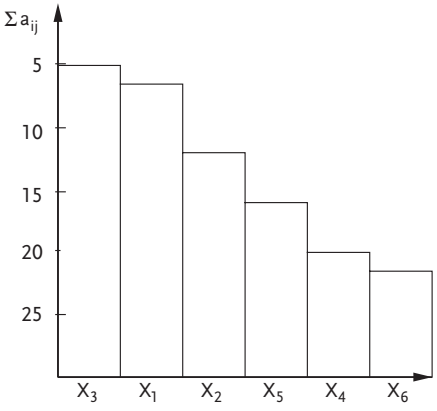

Figure 2.19 Rank histogram

Example 2.9 [12]

A system consisting of a column and a cooler, produces a material characterized by density as a system response. The density of the observed product is affected by six factors: X_1 chlorine consumption; X_2 water consumption in the column; X_3 phlegmatizer consumption; X_4 temperature in column; X_5 level of liquid in column and X_6 water consumption in cooler. The opinions of four researchers are given in Table 2.13. Check the concordance of the researchers' opinions.

Table 2.13 Results of ranking factors

Researchers m	a_{ij}						$T_j = \sum \left(t_j^3 - t_j\right)$
	X_1	X_2	X_3	X_4	X_5	X_6	
1	1.5	5	1.5	4	3	6	2^3-2=6
2	2	3	1	4.5	4.5	6	2^3-2=6
3	2	3	1	5.5	5.5	4	2^3-2=6
4	1.5	3.5	1.5	5	3.5	6	$(2^3-2+2^3-2)=12$
$\sum_1^m a_{ij}$	7	14.5	5	19	16.5	22	$\sum T_j=30$
Δ_i	-7	0.5	-9	5	2.5	8	
$(\Delta_i)^2$	49	0.25	81	25	6.25	64	$\sum_1^6 (\Delta_i)^2 = 225.5$

$$\omega = \frac{12\times 225.5}{4^2\left(6^3-6\right)-4\times 30} \approx 0.805; \qquad \chi_R^2 = \frac{12\times 225.5}{4\times 6(6+1)-\frac{1}{6-1}\times 30} = 16.1$$

Since the tabular value is χ_T^2=11.07 for α=0.05 and f=6-1=5, the hypothesis on the concordance of the researchers' opinions is accepted. A histogram of sums of ranks is shown in Fig. 2.19.

The rank histogram shows that the sum of ranks does not change evenly, so that we can accept the solution to include the following four factors into the basic design of experiment: X_3, X_1, X_2, and X_5. A more cautious approach to drawing conclusions suggests a more detailed check of all six factors in an active experiment for screening factors such as, the *method of random balance*.

■ **Problem 2.1**

A study about the effect of twelve factors of material preparation with regard to fiber tensile strength was done in the textile industry. Four researchers were asked about the prior ranking of the factors. The rank matrix obtained on the basis of a research poll is shown in Table 2.14. Check the concordance of researchers' opinions and choose the significant factors for the next step of experimental research.

Table 2.14 Results of ranking of the factors

Researchers m	a_{ij}												$T_j=\Sigma t_j^3-t_j$
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	
1	8	10.5	10.5	10.5	1	2.5	2.5	10.5	5	4	7	6	**60+6=66**
2	8	9	10	11	1	6.5	6.5	12	2	3	4	5	**8-2=6**
3	6	7.5	7.5	11	2	4.5	4.5	12	1	3	9.5	9.5	**6+6+6=18**
4	7	4	8	10.5	2	10.5	10.5	10.5	1	3	5.5	5.5	**60+6=66**
$\sum_1^m a_{ij}$	29	31	36	43	6	24	24	45	9	13	26	26	$\sum_1^4 T_j = 156$
Δ_i	3	5	10	17	-20	-2	-2	19	-17	-13	0	0	
$(\Delta_i)^2$	9	25	100	289	400	4	4	361	289	169	0	0	$\sum_1^{12} (\Delta_i)^2 = 1650$

Problem 2.2

In a process of chlorinating titanium, seven technological factors were suggested to be analyzed. Five researchers were asked prior to ranking of the factors. The rank matrix is shown in Table 2.15. Determine the concordance coefficient for the researchers' opinions.

Table 2.15 Results of ranking of factors

Researchers m	a_{ij}							T_j
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	
1	1	2	6	4	7	3	5	
2	1	2	7	6	3	5	4	
3	7	1	6	4	2	5	3	
4	3	1	5	6	4	7	2	
5	1	2	6	4	5	7	3	
$\sum_1^m a_{ij}$	13	8	30	24	21	27	17	$\sum T_j=0$
Δ	-7	-12	10	4	1	7	-3	
$(\Delta_i)^2$	49	144	100	16	1	49	9	**S=368**

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Problem 2.3 [16]

The selection of factors from the work [16] was done by the significance of their effects on caoutchouc drying and after processing the opinions of eighteen researchers. The analysis included these eleven factors:: X_1 inlet and outlet moisture ratio; X_2 pH value in the sixth apparatus; X_3 pH value in the seventh apparatus; X_4 NaCl consumption; X_5 serum consumption in the sixth apparatus; X_6 serum consumption in the seventh apparatus; X_7 serum temperature; X_8 latex type; X_9 fat content in caoutchouc; X_{10} latex fat consumption on machine and X_{11} quantity of latex on the surface. Outcomes of the ranking of the factors are shown in Table 2.16. Determine the concordance coefficient and check its significance.

Table 2.16 Results of ranking of factors

Researchers m	a_{ij}											t_j
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	
1	2	5.5	5.5	5.5	8.5	8.5	10.5	2	2	5.5	10.5	**3+4+2+2**
2	1	8	3	6	11	10	7	2	9	5	4	**0**
3	1	4	7	8	6	10	11	3	5	2	9	**0**
4	3	9	1	4	9	9	5	9	6	2	9	**5**
5	9	11	1.5	6	7.5	7.5	3	10	4	1.5	5	**2+2**
6	2.5	2.5	8.5	6	6	8.5	10.5	2.5	6	2.5	10.5	**4+2+3+2**
7	2	2	6	6	6	9	10.5	2	6	6	10.5	**3+5+2**
8	1	2.5	6	6	6	6	10	2.5	6	10	10	**2+5+3**
9	1.5	5.5	9	5.5	9	9	9	3.5	9	1.5	3.5	**2+2+2+5**
10	1	7	7	2	7	7	7	3.5	3.5	10	11	**2+5**
11	2	5.5	5.5	7	9	9	9	3	4	1	11	**2+3**
12	5.5	2.5	10	8	5.5	11	2.5	2.5	8	2.5	8	**4+2+3**
13	5	2.5	9.5	7.5	9.5	11	2.5	1	5	5	7.5	**3+2+2+2**
14	5	3.5	6.5	3.5	9	10	8	1.5	6.5	1.5	11	**2+2+2**
15	2	1	9	5	7	8	10	3	4	6	11	**0**
16	1.5	7	4	7	10	10	10	3	5	1.5	7	**2+3+3**
17	1	4	10	9	7	8	6	2	5	3	11	**0**
18	5.5	5.5	2	3.5	8.5	8.5	8.5	1	3.5	8.5	11	**2+2+4**
$\sum_{1}^{18} a_{ij}$	51.5	98.5	111	105.5	141.5	160	140.5	57	97.5	75.5	160.5	$\sum_{1}^{18} T_j = 946$

Problem 2.4 [14]

Sixteen factors were defined in the process of refining petroleum oils. By the method of prior ranking, determine the concordance coefficient and check its significance. Concordance of data from reference literature is checked based on ranking outcomes shown in Table 2.17.

Table 2.17 Results of ranking of factors

R. m	a_{ij}															
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}	X_{16}
1	1	2	1	1	2	1	1	2	2	4	3	2	4	5	5	3
2	1	4	1	1	3	1	1	2	2	4	3	2	4	4	5	3
3	1	2	5	1	3	1	5	4	4	4	3	4	3	3	1	4
4	2	2	2	2	3	2	1	2	4	2	3	3	2	2	3	4
5	2	3	2	2	2	2	1	2	3	2	1	3	4	3	3	4
6	2	2	2	1	2	1	3	2	2	2	3	2	2	2	2	4
7	3	3	2	2	3	2	1	3	2	4	2	3	3	2	4	3
8	3	1	4	3	2	2	2	3	3	3	4	3	3	3	4	3
9	1	2	4	4	2	4	4	2	3	3	3	4	3	4	4	4
10	2	2	4	4	3	2	3	2	2	4	2	4	5	4	3	5
11	1	3	2	2	4	2	5	4	4	2	4	3	2	4	3	3
12	2	1	5	4	2	5	4	3	2	3	3	5	5	3	3	4
13	3	1	5	4	3	5	4	3	4	2	2	5	3	3	5	4
14	2	2	5	2	2	2	1	3	4	2	4	5	3	4	3	4
15	2	2	5	3	3	3	3	5	2	2	3	4	4	3	4	3
16	1	1	4	2	2	4	3	2	3	3	3	4	3	3	3	3
17	3	1	4	3	2	5	3	2	4	3	3	3	2	3	3	3
$\sum_{1}^{17} a_{ij}$	32	34	37	39	42	44	45	46	48	50	51	62	55	56	58	63	ΣT_j=615
Δ_i	16	14	11	9	6	4	3	2	0	2	3	14	7	8	10	15	
$(\Delta_i)^2$	256	196	121	81	36	16	9	4	0	4	9	196	49	64	100	225	S=1366

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2.2.2 Active Screening Experiment-Method of Random Balance

The application of screening experiments is obligatory when operating with a relatively large number of factors (k≥7), because in the first phase, it facilitates the inclusion of all those factors that do not affect the response greatly. Thus, they also considerably simplify the research of the *factor space-domain* and the modeling of the *response surface*. An active selective method, which may be applied in solving this problem is the *analysis of variance*.

Analysis of variance, as has been said in Sect. 1.5, is based on the fact that the significant effects of certain factors depends on their contribution to the response variance. The analysis of variance is in practice less frequently used in cases of a

large number of factors for it requires a relatively large number of design points-trials.

The efficiency of active screening experiments may be greatly improved by using the *method of random balance*. This method facilitates a relatively simple selection of significant factors above the *noise level* or the level of random response variation, in a limited number of design points. The method is based on the same principle as analysis of variance, i.e. on the fact that the significance of certain factor effects depends of their contribution to the response variance [15]. The selection of factors by significance of their effect on response is realized by the method of random balance in two phases: in the first phase the *matrix of design of experiment* is defined, the experiment takes place and based on its results *scatter diagrams* are constructed. In the second phase, significant factors are taken from scatter diagrams, and their selection is proved by calculations familiar from analysis of variance [15].

Constructing the design of experiments matrix is preceded by coding the factors, the selection of variation levels and by determining the experiment center. When choosing factor-variation levels one should take care for them to be upper and lower limits of normal production or factor variation limit values in the domain of the actor. Factor coding, choice of variation levels and determining the center of the experiment are done by the rules valid for all methods of design of experiments. By the random balance method, factors are mostly varied on two levels (+1;-1), although varying on more than two levels is possible. The number of design points in a matrix is defined so as to be divisible by two. This property simplifies calculations and facilitates estimating linear effects in all cases.

A design matrix by the method of random balance may be constructed in two ways:

- by random distribution of variation levels (+;-) by columns with the help of the random numbers table;
- by random mixing of rows of *regular fractional replicas* of factorial experiments.

The second method of constructing a design matrix is much more widespread, while the first method of *pure random balance* is less efficient and used less frequently. When using fractional replicas one can profit from *semireplicas* from a full factorial experiment. A semireplica may be used for one half of the factors directly, while for the other half, levels are determined by a random choice of rows from the same semi replica. The factors in a random balance matrix are distributed in such a way that significant factors in concordance with prior information or the method of prior ranking of factors, are in the first part of the matrix. This principle may in some cases reduce the number of design points in the next phases, especially if after applying the method of random balance we try to find the optimum. After constructing the matrix, a check should be done in this way:

- the matrix is correct provided there are no correlated columns in it, i.e. the marks in two different columns coincide or do not coincide;
- a matrix must not have columns whose scalar product with any other column gives a column with the same signs.

Processing the obtained results is done after performing the experiment by the constructed matrix. As it has been said, the outcomes are analyzed by scatter diagrams for each factor. Outcomes of the design point for each factor on the diagram are inserted into upper (+) and lower (-) factor levels for all design points of the experiment. The scatter diagram number corresponds to the number of factors. Each factor effect is considered independently from others and is at first determined visually, based on the differences of the response means in the upper and lower levels. The *median* is taken as the response mean at the associated level. Besides, based on the number of so-called *division points* in the upper and lower levels of the scatter diagram, one may judge the significance of factor effects on response. The existence of a large enough number of division points is a strong reason to separate the significant factor, even in cases when the difference between median is small [15]. Visually selected effects are estimated quantitatively by means of *auxiliary tables with several inputs*. In principle, those are the tables with two or three inputs, so that all the necessary mark combinations usually are within the random balance matrix. Construction of auxiliary tables and the calculation of associated effects are identical to the analysis of variance. By dividing the obtained effects by two, the associated regression factor coefficients (b_i) are estimated. The significance of obtained effects is checked by the Students t-criterion along with the corresponding threshold or significance level. Having selected significant factors we correct the initial design point results so as to annul the chosen effects. The response value with a changed sign is added to all response values that correspond to (+) level of the chosen effect. This, in fact, is good for annulling median differences for the levels of the observed factor. Following this correction, the procedure is repeated by including factor interactions. The moment to stop screening factors depends on the Fisher criterion:

$$F = S_R^2 \Big/ S_Y^2 \tag{2.33}$$

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where:

- S_R^2 is value variance of all design points;
- S_Y^2 is variance of system reproducibility as determined by outcomes of replicated experiment design points-trials.

Variance S_R^2 is determined after each result correction, and then the associated value of *Fisher's* criterion is calculated and compared to the tabular value for the chosen significance level. Selection of a factor is stopped at the moment when the variance of the corrected response values is statistically much smaller than the reproducibility system variance. The following examples will demonstrate construction of a design matrix for the method of random balance, by random mixing of kinds of regular fractional replicas or of a full factorial experiment. The procedure consists of breaking all factors into groups that function as research subject physics or as results of prior ranking of the factors. Groups should thereby consist of not more than five to six factors. Design matrix of *full factorial experiment*[1)] or *fractional factorial experiment*[2)] corresponds to each group, with types of a random order. A better

1) Full factorial experiment FUFE

2) Fractional factorial experiment FRFE

solution is the grouping with three-four factors and the correspondence of the *FUFE* matrix.

Example 2.10 [17]

Filtration conditions were studied in a process of producing dyes. The filtration phenomenon is expressed with these four factors:

X_1 concentration of a solution being filtered;
(+) upper level-, concentrated solution;
(-) lower level, diluted solution;

X_2 time of dosing solution;
(+) upper level, fresh solution;
(-) lower level, old solution;

X_3 contents of filler in solution;
(+) upper level, filler present;
(-) lower level, filler not present;

X_4 temperature of solution;
(+) upper level, higher temperature;
(-) lower level, lower temperature.

Following the procedure of the matrix construction, the factors are divided into two groups of two factors each. The first group consists of X_1 and X_2, and the other one of X_3 and X_4 factors. A *FUFE* matrix has four design points for two factors and is shown in Table 2.18.

Table 2.18 FUFE

Number	Factors	
D.P.	X_1	X_2
1	−	−
2	+	−
3	−	+
4	+	+

Table 2.19 Matrix of random balance

Number	Factors				Response
D.P.	X_1	X_2	X_3	X_4	Y
1	+	−	−	+	114
2	−	−	+	+	106
3	−	+	+	−	120
4	+	+	−	−	132

By random choice from matrix *FUFE* or Table 2.18, we take for the first group of 2., 1., 3. and 4., row respectively. For the second group of factors we take in the same way 3., 4., 2. and 1. row from the *FUFE* matrix. In this way, the design matrix for the random balance method is constructed. In this example, the response is represented by the product purity y.

The values for product purity shown in Table 2.19 were obtained after the experiment. Scatter diagrams are drawn from the response value for each factor. The scatter diagram for all four factors is shown in Fig. 2.20.

The median is determined on this diagram for each factor level. When the number of points in one level is odd 2n+1, then the median is marked in the point n+1. If the number of points is even 2n, the median passes through the point that is the

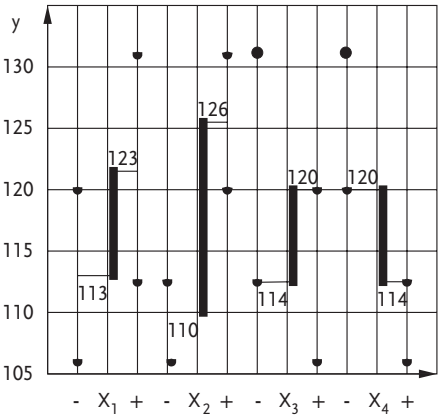

Figure 2.20 Scatter diagram

arithmetic average between n and n+1 points. The difference between the upper and lower level medians, gives the effect of the associated factor:

$$\begin{aligned} E_{X_1} &= 123 - 113 = +10 \\ E_{X_2} &= 126 - 110 = +16 \\ E_{X_3} &= 120 - 114 = +6 \\ E_{X_4} &= 114 - 120 = -6 \end{aligned} \tag{2.34}$$

Due to the calculated effects, the significant factors after the first screening are X_1 and X_2.

These factors are then quantitatively checked by forming an auxiliary table with two inputs:

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Table 2.20 Table with two inputs

	$+X_1$	$-X_1$
$+X_2$	132	120
	...	...
	$\bar{y}_1 = 132$	$\bar{y}_2 = 120$
$-X_2$	114	106
	...	...
	$\bar{y}_3 = 114$	$\bar{y}_4 = 106$

These effects of selected factors are obtained from Table 2.20:

$$\begin{aligned} E_{X_1} &= (+X_1) - (-X_1) = \frac{\bar{y}_1 + \bar{y}_3}{2} - \frac{\bar{y}_2 + \bar{y}_4}{2} = +10 \\ E_{X_2} &= (+X_2) - (-X_2) = \frac{\bar{y}_1 + \bar{y}_2}{2} - \frac{\bar{y}_3 + \bar{y}_4}{2} = +16 \end{aligned} \tag{2.35}$$

To obtain an answer to the question whether other factors are significant too, it is necessary to exclude effects of selected factors and then repeat the procedure. The effects of selected factors are analyzed in such a way that in each design point where X_1 is in the upper level of (+) response value factor effect X_1 is added with a changed sign (E_{X_1} =-10), and where X_2 is in the upper level of (+) response value E_{X_2} is added with a changed sign (E_{X_2} =-16). In this way, Table 2.21 is obtained with corrected responses.

Table 2.21 Design matrix with corrected responses

Number d.p.	Factors				Responses	
	X_1	X_2	X_3	X_4	y	y′
1	+	−	−	+	114	104
2	−	−	+	+	106	106
3	−	+	+	−	120	104
4	+	+	−	−	132	106

If we now reconstruct the scatter diagram we will see that effects for factors X_3 and X_4 are irrelevant and close to zero. The conclusion is that factors X_3 and X_4 are insignificant. Besides selecting factors by their significance, it is necessary to single out the interaction effects. Signs for associated interactions are obtained by simply multiplying the signs of associated factors, as shown in Table 2.22.

Table 2.22 Design matrix with interactions

Number trials	Factors				Interactions						Response y
	X_1	X_2	X_3	X_4	X_1X_2	X_1X_3	X_1X_4	X_2X_4	X_2X_3	X_3X_4	
1	+	−	−	+	−	−	+	−	+	−	114
2	−	−	+	+	+	−	−	−	−	+	106
3	−	+	+	−	−	−	+	−	+	−	120
4	+	+	−	−	+	−	−	−	−	+	132

Only *even interactions* are analyzed, since higher-order interactions are less probable. Drawing scatter diagrams for all even interactions is a troublesome job. Scatter diagrams are therefore constructed for those even interactions for which there is an indication that they may be crucial. An indication is that higher effects are obtained from interactions where point dispersion of factors in upper and lower levels is reversed. Scatter diagram of even interactions is obtained in the same way as for the factors and is shown in Fig. 2.21.

Interaction effects X_1X_3, X_2X_3 and X_2X_4 are singled out from the scatter diagram. An auxiliary table, Table 2.23, with three inputs is formed for the given interactions. Vacancies in this table indicate that interaction columns X_1X_3 and X_2X_4 are intercor-

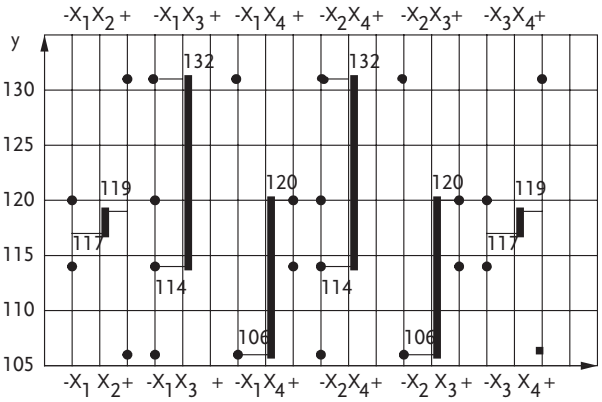

Figure 2.21 Scatter diagram

related. This is also proved by the equality of signs, which does not allow actual calculation of the effects. Table 2.24 with two inputs is therefore formed with interaction columns X_1X_3 and X_2X_4 left vacant. In a larger number of design points and for more factors, column intercorrelation is hardly probable. Finally, we may conclude that by the method of random balance these significant factors and interactions were singled out:

- factor X_1 with effect E_{X1}=+10;
- factor X_2 with effect E_{X2}=+16;
- even interaction X_2X_4;
- even interaction X_1X_3.

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Table 2.23 Table with three inputs

	$+X_1X_3$		$-X_1X_3$	
	$+X_2X_4$	$-X_2X_4$	$+X_2X_4$	$-X_2X_4$
$+X_1X_3$	...	...	...	114
	...	...	...	120
	132	...	...	...
	$\bar{y}_1 = 132$			$\bar{y}_4 = 117$
$-X_1X_3$	...	...	...	106
	...	...	...	...
	...	...	...	...
				$\bar{y}_8 = 106$

Table 2.24 Table with two inputs

	$+X_2X_4$	$-X_2X_4$
$+X_1X_3$	132	...
	...	...
	$\bar{y}_1 = 132$	...
$-X_1X_3$	...	114
	...	106
	...	120
		$\bar{y}_4 = 113$

Interaction effects X_1X_3 and X_2X_4 could not be singled out due to column correlation, so they remained *mixed*.

This example, too, has shown the advantage of constructing the design matrix for the method of random balance from *FUFE* and *FRFE*, since for the two obtained factors X_1 and X_2 we already have a finished experiment by a full factorial design. These results are for all four design points of the design in Table 2.19. Since the method of random balance also gives the effects, they may be divided by two to obtain regression coefficients for a robust approximation of response:

$$\widehat{y} = \bar{y} + 10/2 \times X_1 + 16/2 \times X_2 = (114 + 106 + 120 + 132)/4 + 5X_1 + 8X_2$$

$$\widehat{y} = 118 + 5X_1 + 8X_2\,;\,(-1 \le X_1 \le 1\,;\,-1 \le X_2 \le 1) \tag{2.36}$$

The following could be said, after all things disclosed, about the active method of screening factors or the method of random balance:

- the method of random balance is effective in complicated, unclear situations when fast screening of factors is necessary; the method is the more effective the fewer significance factors exist, since an increase in their number increases the design-point variance;
- the method of random balance is more of a new approach to an experiment then the processing of experiment data;
- even untrained researchers may profit from the method, even in situations with a large number of factors;
- the method facilitates singling out significant factors with sufficient confidence level;
- the method of random balance is primarily meant for researchers in labs and pilot-plants since in full-scale plants it requires disrupting of the current process conditions.

Advantages and disadvantages of the method are shown in Table 2.25:

Table 2.25 Advantages and disadvantages of random balance method

No.	Advantages	Disadvantages
1	Possibility to include many factors, decrease in risk to omit a factor	Unclear determination of risk and reduction of chance to omit a factor
2	Assumes exponential ranking of factors, which seems natural	Difficult to expect exponential distribution of factors
3	Random design, simple designing is used, experiment are less strict	System designs, standard designs, orthogonality are all better
4	Few design points necessary, designs oversaturated	Not enough information, effect estimates mixed
5	Error slightly bigger but the problem is screening most significant factors	Less significant factors are hard to detect
6	Simple graphic analysis	It is present with other methods
7	Possibility to vary factors on several levels	This complicates interaction
8	Numerous examples of application	No theoretical ground dangerous to recommend it
9	Simple model	More complex model needed

For practical application several prepared design matrixes by the method of random balance [18]–[20] are recommended.

Table 2.26 k=8

No. D.P.	Factors							
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8
1	−	−	+	+	−	+	−	+
2	+	+	+	+	+	−	−	+
3	−	+	+	−	+	−	+	−
4	+	−	−	+	+	+	+	+
5	−	+	−	+	−	−	−	−
6	+	+	−	−	−	−	+	+
7	−	−	−	−	−	+	+	−
8	+	−	+	−	+	+	−	−

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Table 2.27 k=10

No. D.P.	Factors									
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}
1	−	−	−	−	−	−	−	−	+	+
2	+	+	−	−	−	+	+	−	−	−
3	+	−	+	+	+	+	−	+	−	−
4	−	+	+	+	+	−	−	−	−	−
5	+	−	+	−	−	+	−	−	+	−
6	−	+	+	−	−	+	+	+	−	+
7	−	−	−	+	+	+	−	−	−	+
8	+	+	−	+	+	+	−	+	+	+
9	+	−	−	−	+	−	+	+	−	−
10	−	+	−	−	+	−	+	−	+	−
11	−	−	+	+	−	−	+	+	+	+
12	+	+	+	+	−	−	−	+	−	−
13	+	−	−	+	−	+	+	−	+	+
14	−	+	−	+	−	−	−	+	+	−
15	−	−	+	−	+	−	+	−	−	−
16	+	+	+	−	+	+	+	+	+	−

Table 2.28 k=5

No. D.P.	Factors				
	X_1	X_2	X_3	X_4	X_5
1	+	+	+	−	−
2	+	+	−	−	+
3	+	−	+	+	−
4	+	−	−	+	+
5	−	+	+	+	+
6	−	+	−	+	−
7	−	−	+	−	+
8	−	−	−	−	−

Table 2.29 k=12

No. D.P.	Factors											
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}
1	−	+	−	+	+	+	+	−	+	+	+	+
2	−	−	+	−	−	−	−	−	+	+	+	−
3	−	+	+	−	+	−	−	−	−	−	+	+
4	+	−	−	−	+	+	−	−	−	+	−	−
5	+	−	−	+	−	−	−	+	−	+	+	+
6	−	+	−	−	+	−	−	−	+	−	−	−
7	+	+	−	−	−	+	+	−	+	−	−	+
8	−	−	+	+	+	−	−	+	+	+	−	−
9	−	−	+	+	−	−	+	+	+	−	+	+
10	−	+	+	−	−	−	+	+	+	+	+	−
11	+	+	−	+	+	+	−	−	−	+	−	+
12	−	−	−	−	−	+	+	+	−	+	+	−
13	+	+	+	+	+	+	−	−	−	+	−	+
14	−	−	−	+	−	+	+	−	−	−	−	−
15	−	−	−	+	−	+	−	+	+	−	−	+
16	+	−	−	−	+	−	−	−	+	+	+	−
17	+	+	+	+	−	+	−	+	+	+	−	+
18	+	−	+	−	+	−	+	+	−	−	−	−
19	+	−	+	−	−	−	+	+	−	−	+	+
20	−	+	−	+	−	+	+	+	+	+	−	−
21	−	+	+	−	+	+	+	−	−	−	+	+
22	+	−	+	+	+	−	−	−	+	−	+	−
23	+	+	+	+	−	+	+	+	−	−	−	+
24	+	+	−	−	+	−	+	+	−	−	+	−

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Table 2.30 k=14

No. D.P.	Factors													
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}
1	−	−	+	+	+	−	+	−	+	−	+	+	−	+
2	+	−	−	+	−	−	−	−	−	+	−	−	−	+
3	−	+	−	+	+	−	−	+	+	+	+	−	+	−
4	+	+	+	+	+	+	−	−	+	+	−	+	−	+
5	−	−	−	−	−	+	+	+	−	−	+	−	−	−
6	+	−	+	−	+	+	+	−	+	+	+	−	+	+
7	−	+	+	−	−	+	+	−	+	+	−	+	+	−
8	+	+	−	−	−	−	−	+	−	−	+	−	−	−
9	−	−	+	−	−	+	−	+	−	+	+	+	+	+
10	+	−	−	−	−	+	−	−	−	−	−	−	+	+
11	−	+	−	−	+	−	+	−	+	−	−	−	−	−
12	+	+	+	−	+	−	−	−	−	−	−	−	+	−
13	−	−	−	+	+	+	−	+	−	+	+	+	+	−
14	+	−	+	+	−	−	+	+	+	−	+	+	−	−
15	−	+	+	+	−	−	+	+	+	+	−	+	+	+
16	+	+	−	+	+	+	+	+	−	−	−	+	−	+

Table 2.31 k=19

No. D.P.	Factors																		
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}	X_{16}	X_{17}	X_{18}	X_{19}
1	−	−	+	+	+	−	+	+	+	−	+	−	−	−	−	−	−	−	−
2	+	−	−	+	+	−	+	+	−	−	+	+	−	+	+	−	+	+	−
3	−	+	−	+	+	+	−	+	+	+	+	−	+	−	−	−	+	+	+
4	+	+	+	+	+	−	+	−	+	−	+	+	+	−	+	+	−	+	−
5	−	−	−	−	+	+	−	+	+	+	−	−	−	+	+	+	+	−	+
6	+	−	+	−	+	−	+	−	−	−	+	−	−	+	+	−	−	+	+
7	−	+	+	−	+	−	+	+	−	−	−	+	+	−	−	−	+	+	−
8	+	+	−	−	+	+	−	−	−	−	+	−	+	−	+	+	+	−	−
9	−	−	+	−	−	−	−	+	+	+	−	−	+	−	+	+	+	−	+
10	+	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−	+
11	−	+	−	−	−	+	+	+	+	+	+	+	−	+	+	−	−	+	−
12	+	+	+	−	−	−	−	+	−	+	+	−	+	+	−	+	−	−	−
13	−	−	−	+	−	+	+	−	+	−	−	+	+	+	−	+	−	+	+
14	+	−	+	+	−	−	−	−	+	+	+	−	−	−	−	+	+	−	−
15	−	+	+	+	−	+	+	+	−	+	+	+	−	−	+	+	−	+	+
16	+	+	−	+	−	+	+	+	+	+	−	+	−	−	+	−	+	−	+
17	−	−	+	+	−	+	−	+	−	−	+	−	+	+	−	−	+	−	−

Table 2.31 Continued

No. D.P.	Factors																		
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}	X_{16}	X_{17}	X_{18}	X_{19}
18	+	−	−	+	−	+	−	−	−	+	+	+	+	+	+	−	−	−	−
19	−	+	−	+	−	+	−	−	−	−	+	+	−	−	+	−	+	+	+
20	+	+	+	+	−	−	+	−	+	−	−	+	−	−	+	+	−	−	−
21	−	−	−	−	−	−	+	−	−	−	−	−	+	−	−	+	+	+	−
22	+	−	+	−	−	+	−	+	−	−	−	−	−	+	−	+	−	+	+
23	−	+	+	−	−	+	−	−	−	+	−	−	−	−	+	−	+	+	+
24	+	+	−	−	−	+	+	+	−	+	−	+	+	+	+	−	−	+	−
25	−	−	+	−	+	−	+	+	−	+	+	−	−	+	−	+	+	−	−
26	+	−	−	−	+	−	+	+	+	−	+	+	+	+	−	−	+	−	+
27	−	+	−	−	+	+	−	−	+	+	−	+	−	+	−	+	+	+	+
28	+	+	+	−	+	−	+	−	−	−	−	−	+	+	−	−	−	+	+
29	−	−	−	+	+	+	−	−	+	+	+	+	+	−	−	−	−	−	+
30	+	−	+	+	+	−	−	+	+	+	−	−	+	+	−	+	−	−	+
31	−	+	+	+	+	−	−	−	+	−	−	+	−	+	+	+	−	+	−
32	+	+	−	+	+	+	+	−	+	+	−	+	+	−	+	+	+	−	+

It should be noted that when doing an experiment by the method of random balance, it is not necessary to have a random order of doing design points, since the randomization principle has been introduced in constructing the matrix.

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Example 2.11

Adhesion on "HLORIN"-type fibers, has been studied as a function of five process factors. The names of factors, with their variation levels, are shown in Table 2.32. Matrix 2^3 of full factorial experiment has been used in constructing random balance matrix. The design matrix by the method of random balance with experimental results is shown in Table 2.33. Note that each design point was repeated 20 to 50 times due to high non reproducibility of the system.

Table 2.32 Variation levels

Factors	Levels	
	−	+
X_1 pressing temperature °C	140	170
X_2 pressing pressure kp/cm^2	5	20
X_3 time of pressing min	0.5	2.5
X_4 time of preprocessing min	1	3
X_5 fiber nature	Viscose	Capronate

Table 2.33 Design matrix

No. D.P.	Factors					Response $\bar{y}$
	X_1	X_2	X_3	X_4	X_5	
1	+	+	+	−	−	32.6
2	+	+	−	−	+	15.2
3	+	−	+	+	−	18.9
4	+	−	−	+	+	15.2
5	−	+	+	+	+	14.6
6	−	+	−	+	−	33.1
7	−	−	+	−	+	14.0
8	−	−	−	−	−	24.5

Scatter diagram was constructed based on the results from Table 2.33. The effects of factors X_2 and X_5 were visually screened out from the scatter diagram shown in Fig. 2.22

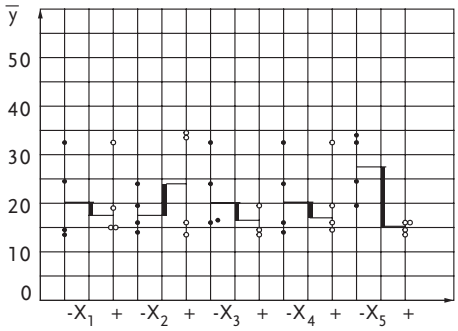

Figure 2.22 Scatter diagram

Table 2.34 is formed for the two separated factors with two inlets. Effects of factors X_2 and X_5 are quantitatively determined from the table. Then, a significance check of obtained effects is done by the *Students* t-criterion.

Table 2.34 Table with two inputs

	$+X_2$		$-X_2$	
$+X_5$	15.2	$\sum y_1 = 29.8$	15.2	$\sum y_2 = 29.2$
	14.6	$\bar{y}_1 = 14.9$	14.0	$\bar{y}_2 = 14.6$
$-X_5$	33.1	$\sum y_3 = 65.7$	24.5	$\sum y_4 = 43.4$
	32.6	$\bar{y}_3 = 32.9$	18.9	$\bar{y}_4 = 21.7$

$$E_{X_5} = \frac{\bar{y}_1+\bar{y}_2}{2} - \frac{\bar{y}_3+\bar{y}_4}{2} = \frac{14.9+14.6}{2} - \frac{32.9+21.7}{2} = -12.52$$

$$E_{X_2} = \frac{\bar{y}_1+\bar{y}_3}{2} - \frac{\bar{y}_2+\bar{y}_4}{2} = \frac{14.9+32.9}{2} - \frac{14.6+21.7}{2} = +5.72$$

Significance check of the effects:

$$t_{X_5} = \frac{(\bar{y}_1+\bar{y}_2)-(\bar{y}_3+\bar{y}_4)}{S_R\sqrt{\sum\frac{1}{n_i}}} = \frac{14.9+14.6-32.9-21.7}{\sqrt{8.52}} = -8.57$$

$$t_{X_2} = \frac{(\bar{y}_1+\bar{y}_3)-(\bar{y}_2+\bar{y}_4)}{S_R\sqrt{\sum\frac{1}{n_i}}} = \frac{14.9+32.9-14.6-21.7}{\sqrt{8.52}} = +3.92$$

where:

$\bar{y}_1;\bar{y}_2;\bar{y}_3;\bar{y}_4$ are response means by cells of auxiliary table;

S_R^2 is response variance by cells;

n_i is number of design points in each cell.

The value $\sum S_R^2 \Big/ n_i$ is obtained by calculations shown in Table 2.35.

Table 2.35 Calculation of variance S_R^2

Number of cells	n_i	$\sum y_i$	$(\sum y_i)^2$	$\sum y_i^2$	$S_R^2 = \frac{\sum y_i^2}{n_i-1} - \frac{(\sum y_i)^2}{n_i(n_i-1)}$	$\frac{S_R^2}{n_i}$
1	2	29.8	888.04	444.2	016	0.08
2	2	29.2	852.64	427.4	1.08	0.54
3	2	65.7	4316.49	2158.4	0.12	0.06
4	2	43.4	1883.56	957.5	15.68	7.84
$\sum$	8					8.52

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The number of degrees of freedom is the difference between the total number of experimental design points and the number of cells, in this case it is:

f=8-4=4.

For the threshold or significance level α=0.05 and for f=4 we have the tabular value of *Students* criterion t_T=2.78. Since the calculated values are above The tabular values for the T-test, the separated effects are statistically significant with 95% confidence. The next step was correction of response values for -5.72 and +12.52 in those design points where X_2 and X_5 are in the upper levels. After a response correction a new scatter diagram was constructed for the factors and even interactions. X_3 and X_2X_5 were visually separated with their effects -5.42 and -1.98, respectively. The significance check of these effects showed that X_2X_5 had 95% and X_3 90% confidence levels. After the correction of corrected responses with the effects of factors X_3 and X_2X_5, the variance of twice-corrected responses S_R^2=1.27, approached the reproducibility var-

iance S_R^2=0.50. A check of Fisher's criterion offered the value F_R=1.27/0.50=2.54. Since the tabular value is $F_{0.05}$=2.36, further process of screening factors is stopped.

Table 2.36 Summarized results

Screening phase	Visual screening	Effect values	t_R	b_i
Initial response values	X_5	-12.52	8.57	-6.26
	X_2	+5.75	3.92	+2.86
First response correction	X_5X_3	-5.42	6.69	-2.71
		-1.98	2.22	-0.99
Second response correction	X_4X_1	-1.97	1.54	-0.54
		-1.13	1.62	-0.57

Hence, the adhesion of fibers is significantly affected by factors X_5; X_2 and X_3. The expected effect of X_1 factor proved to be marginal. This may be explained only by the fact that its value in the center of the performed experiment is either maximal or its variation interval has been badly chosen. Further experiments have proved the first assumption. The summarized result of the method of random balance is shown in Table 2.36.

Example 2.12 [18]

Factors were not screened out in Example 2.8 by the method of prior ranking, so that a matrix of random balance was constructed for all eight factors, Table 2.37. The experiment was done by one replication of the design point in order to establish the variance of system reproducibility.

Table 2.37 Design of experiment

	Factors								Responses		
Basic level	60	50	60	1200	15	15	6	310			
Variation interval	10	30	40	400	5	5	4	305			
Upper level (+)	70	80	100	1600	20	20	10	615			
Lower level (-)	50	20	20	800	10	10	2	5			
Number of design points	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	y_{01}	y_{02}	$\bar{y}$
1	−	−	+	+	−	+	−	+	528.00	540.80	534.40
2	+	+	+	+	+	−	−	+	345.60	345.30	345.45
3	−	+	+	−	+	−	+	−	512.00	521.60	516.80
4	+	−	−	+	+	+	+	+	713.60	576.00	644.80
5	−	+	−	+	−	−	−	−	1067.20	912.00	989.60
6	+	+	−	−	−	−	+	+	870.40	832.00	851.20
7	−	−	−	−	−	+	+	−	1648.00	1657.60	1652.80
8	+	−	+	−	+	+	−	−	572.80	454.40	513.60

Note: S_y^2=3678.89; f=8(2-1)=8

The scatter diagram was constructed from the obtained response values, Fig. 2.23.

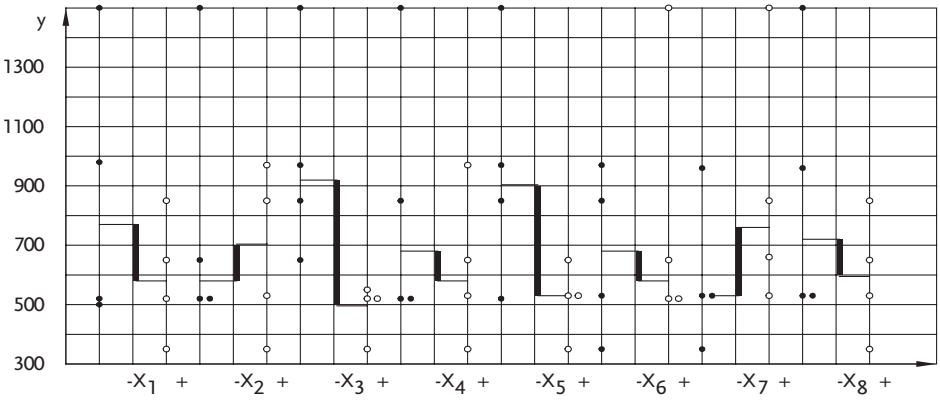

Figure 2.23 Scatter diagram

Based on the scatter diagram factors, X_3 and X_5 are visually screened out. A table with two inputs is formed for these factors. Quantitative effects are calculated from the auxiliary table and then checked by the t-test. Since both effects are significant, the first correction is done and then the procedure is repeated. Factors X_4 and X_8 are visually screened out in the second step. Quantitative calculation of effects and t-test show that only factor X_8 is significant. The effect of this factor is annulled by the second response correction. Now, interactions are introduced into the scatter diagrams so that X_1X_7 and X_5X_8 are visually screened out. Quantitative values of the effects of these two interactions were not statistically important. Since Table 2.38 shows that the variance of corrected responses is significantly close to reproducibility variance, the selection procedure is over. The diagram in Fig. 2.24, which clearly shows a reduction in residual variance (S_R^2), is especially important for the method of random balance.

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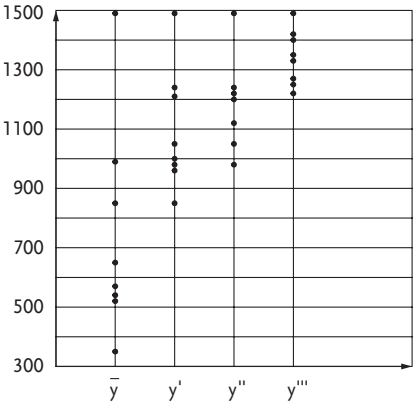

Figure 2.24 Corrected responses

Table 2.38 Corrected responses

No. D.P.	Factors								Corrected responses			
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	$\bar{y}$	y'	y''	y'''
1	−	−	+	+	−	+	−	+	534.40	942.56	1204.85	1428.83
2	+	+	+	+	+	−	−	+	345.45	1051.37	1313.66	1313.66
3	−	+	+	−	+	−	+	−	516.80	1222.72	1222.72	1222.72
4	+	−	−	+	+	+	+	+	644.80	942.56	1204.85	1428.83
5	−	+	−	+	−	−	−	−	989.60	989.60	989.60	1213.58
6	+	+	−	−	−	−	+	+	851.20	851.20	1113.49	1337.47
7	−	−	−	−	−	+	+	−	1652.80	1652.80	1652.80	1652.80
8	+	−	+	−	+	+	−	−	513.60	1219.52	1219.52	1219.52
$\bar{\bar{y}}$									756.08	1109.04	1240.19	1352.18
S_R^2									173411.41	65758.33	36817.62	22484.50
$F = S_R^2/S_y^2$									47.14	17.87	10.01	6.11

Note: S_y^2=3678.89; f=8; Reproducibility variance $F_{T(7;8;0.99)}$=6.18

Example 2.13 [15]

Due to the advantages and disadvantages of the method of random balance, which have been mentioned in this section, a demonstration of efficiency of the method will be given in this example on an artificially constructed problem and where we know, in advance, the effects that should be screened. It will also be shown that, generally speaking, the method of random balance with more than two levels of factor variation has no advantage. A demand for more than two levels is justified only in cases with qualitative factors.

Assume that twelve factors, X_1 to X_{12}, should be screened. The random balance matrix will consist of two independent semi-replicas of a 2^6 full factorial experiment, with rows or design points that are randomly distributed. The 32 design points thus synthesized will start with the values taken from a normal population with the mean 100 and the standard deviation δ_0=2.0. The effects of factors have been introduced in the way that the following values were added to the best values of selected factors in the upper level (+):

value -15 added to factor X_7
value -12 added to factor X_4
value +10 added to factor X_{10} and X_{11}
value +8 added to factor X_1
value +6 added to factor X_5 and X_8
value +4 added to factor X_2
value -4 added to factor X_9

Table 2.39 shows groupings of factors with associated semireplicas of 2^6 full factorial design.

Table 2.39 Semireplica of full factorial design 2^6

First group of factors	X_1	X_2	X_3	X_4	X_5	X_6
Secon group of factors	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}
1	+	+	+	+	+	+
2	+	+	+	+	−	−
3	+	+	+	−	+	−
4	+	+	+	−	−	+
5	+	+	−	+	+	−
6	+	+	−	+	−	+
7	+	+	−	−	+	+
8	+	+	−	−	−	−
9	+	−	+	+	+	−
10	+	−	+	+	−	+
11	+	−	+	−	+	+
12	+	−	+	−	−	−
13	+	−	−	+	+	+
14	+	−	−	+	−	−
15	+	−	−	−	+	−
16	+	−	−	−	−	+
17	−	+	+	+	+	−
18	−	+	+	+	−	+
19	−	+	+	−	+	+
20	−	+	+	−	−	−
21	−	+	−	+	+	+
22	−	+	−	+	−	−
23	−	+	−	−	+	−
24	−	+	−	−	−	+
25	−	−	+	+	+	+
26	−	−	+	+	−	−
27	−	−	+	−	+	−
28	−	−	+	−	−	+
29	−	−	−	+	+	−
30	−	−	−	+	−	+
31	−	−	−	−	+	+
32	−	−	−	−	−	−

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Table 2.40 shows a complete design matrix by the method of random balance; original data taken from normal population y_0; y synthesized response to which values of effects were added and the phases of factor screening with corrected response y^I; y^{II}; y^{III} and y^{IV} and their standard deviations.

The catter diagram, Fig. 2.25, has been constructed for response y. Medians for upper and lower levels of associated factors were used on the same diagrams in

order to select the factors visually. Median or effect value differences were also given on the scatter diagram.

Table 2.40 Design of experiment

No. D.P.	Combinations		Factors												y_0	Responses				
	X_1-X_6	X_7-X_{12}	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}		y	Y^I	y^{II}	y^{III}	y^{IV}
1	18	7	−	+	+	+	−	+	+	+	−	−	+	+	101	78	103	103	103	98
2	8	32	+	+	−	−	−	−	−	−	−	−	−	−	99	103	92	101	96	96
3	4	6	+	+	+	−	−	+	+	+	−	+	−	+	100	97	99	99	99	94
4	6	16	+	+	−	+	−	+	+	−	−	−	−	+	100	77	91	100	95	95
5	3	19	−	+	+	−	+	−	−	+	+	−	+	+	99	113	102	102	97	97
6	23	5	−	+	−	−	+	−	+	+	−	+	+	−	96	77	90	99	94	94
7	17	4	−	+	+	+	+	−	+	+	+	−	−	+	97	66	91	100	95	95
8	26	27	−	−	+	+	−	−	−	−	+	−	+	−	99	93	105	105	100	100
9	31	2	−	−	−	−	+	+	+	+	+	+	−	−	99	86	99	99	94	99
10	15	28	+	−	−	−	+	−	−	−	+	−	−	+	99	93	82	91	91	96
11	28	3	−	−	+	−	−	+	+	+	+	−	+	−	102	87	100	100	100	100
12	16	25	+	−	−	−	−	+	−	−	+	+	+	+	100	100	89	97	92	97
13	30	13	−	−	−	+	−	+	+	−	−	+	+	+	103	72	97	106	101	101
14	11	22	+	−	+	−	+	+	−	+	−	+	−	−	106	124	113	113	108	103
15	27	8	−	−	+	−	+	−	+	+	−	−	−	−	98	83	96	105	100	95
16	19	15	−	+	+	−	+	+	+	−	−	−	+	−	99	88	101	101	101	96
17	14	23	+	−	−	+	−	−	−	+	−	−	+	−	100	96	97	97	97	97
18	20	18	−	+	+	−	−	−	−	+	+	+	−	+	100	100	100	100	100	100
19	22	20	−	+	−	+	−	−	−	+	+	−	−	−	98	72	84	93	93	98
20	32	17	−	−	−	−	−	−	−	+	+	+	+	−	100	86	86	95	95	100
21	25	1	−	−	+	+	+	+	+	+	+	+	+	+	100	69	94	103	98	98
22	2	26	+	+	+	+	−	−	+	−	+	+	−	−	101	103	104	104	99	99
23	1	11	+	+	+	+	+	+	−	−	+	−	+	+	96	77	91	91	91	91
24	7	21	+	+	−	−	+	+	−	+	−	+	+	+	100	104	93	102	102	97
25	21	30	−	+	−	+	+	+	−	−	−	+	−	+	96	84	96	96	96	96
26	12	12	+	−	+	−	−	−	+	−	+	−	−	−	100	89	91	100	95	95
27	24	24	−	+	−	−	−	+	−	+	−	−	−	+	102	92	92	101	101	101
28	10	9	+	−	+	+	−	+	+	−	+	+	+	−	101	78	92	101	96	96
29	5	29	+	+	−	+	+	−	−	−	−	+	+	−	101	87	88	97	97	97
30	9	10	+	−	+	+	+	−	+	−	+	+	−	+	100	81	95	95	95	95
31	13	31	+	−	−	+	+	+	−	−	−	−	+	+	103	99	100	100	100	100
32	29	14	−	−	−	+	+	−	+	−	−	+	−	−	99	72	97	97	97	97
Standard deviation s_r															2.1	13.5	6.6	4.3	3.6	2.6

Factors X_1; X_4 and X_7 with their respective effects E_{X1}=13.0, E_{X4}=14.5 and E_{X7}=19.5 are also screened out visually from the scatter diagram. A statistical significance check of these effects is done by quantitative calculation of their values through a table with three inputs and a check with the Students t-test. An auxiliary table with three inputs is shown in Table 2.41.

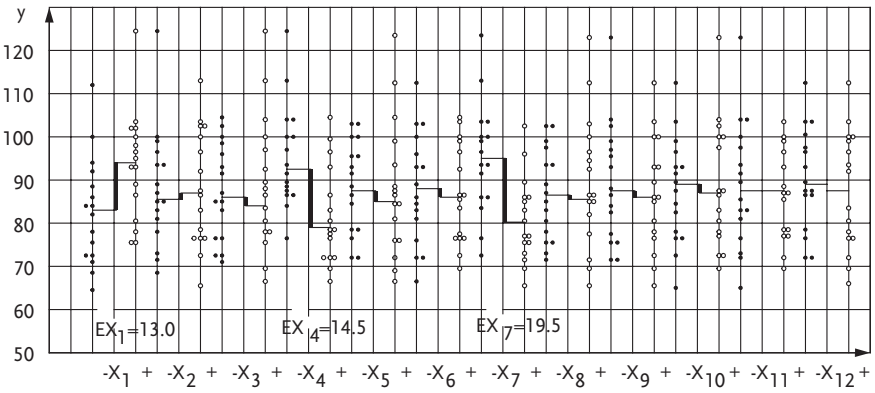

Figure 2.25 Scatter diagram

Table 2.41 Auxilliary table with three inputs

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	-X4	+X4		
-X7	+X7	-X7	+X7	
124	97	103	81	+X1
113	89	99	78	
104		96	77	
103		87	77	
100				
93				
$\sum y_1 = 637$	$\sum y_2 = 186$	$\sum y_3 = 385$	$\sum y_4 = 313$	
$\bar{y}_1 = 106.17$	$\bar{y}_2 = 93.00$	$\bar{y}_3 = 96.25$	$\bar{y}_4 = 78.25$	
100	88	93	78	-X1
92	87	84	72	
86	86	72	72	
	83		69	
	77		69	
$\sum y_5 = 278$	$\sum y_6 = 421$	$\sum y_7 = 249$	$\sum y_8 = 357$	
$\bar{y}_5 = 92.67$	$\bar{y}_6 = 84.20$	$\bar{y}_7 = 83.00$	$\bar{y}_8 = 71.40$	

The effects of factors are calculated thus:

$$E_{X_1} = \frac{\bar{y}_1+\bar{y}_2+\bar{y}_3+\bar{y}_4}{4} - \frac{\bar{y}_5+\bar{y}_6+\bar{y}_7+\bar{y}_8}{4} =$$
$$= \frac{106.17+93.00+96.25+78.25}{4} - \frac{92.67+84.20+83.00+71.40}{4} = +10.60$$

$$E_{X_4} = \frac{\bar{y}_3+\bar{y}_4+\bar{y}_7+\bar{y}_8}{4} - \frac{\bar{y}_1+\bar{y}_2+\bar{y}_5+\bar{y}_6}{4} =$$
$$= \frac{96.25+78.25+83.00+71.40}{4} - \frac{106.17+93.00+92.67+84.20}{4} = -11.87$$

$$E_{X_7} = \frac{\bar{y}_2+\bar{y}_4+\bar{y}_6+\bar{y}_8}{4} - \frac{\bar{y}_1+\bar{y}_3+\bar{y}_5+y_7}{4} =$$
$$= \frac{93.00+78.25+84.20+71.40}{4} - \frac{106.17+96.25+92.67+83.00}{4} = -12.81$$

The check of effects is done by the *Students* t-criterion. Arithmetic values of the t-test are determined from these relations:

$$t_{X_1} = \frac{(\bar{y}_1+\bar{y}_2+\bar{y}_3+\bar{y}_4)-(\bar{y}_5+\bar{y}_6+\bar{y}_7+\bar{y}_8)}{S_R\sqrt{\sum\frac{1}{n_i}}} \tag{2.37}$$

$$t_{X_4} = \frac{(\bar{y}_3+\bar{y}_4+\bar{y}_7+\bar{y}_8)-(\bar{y}_1+\bar{y}_2+\bar{y}_5+\bar{y}_6)}{S_R\sqrt{\sum\frac{1}{n_i}}} \tag{2.38}$$

$$t_{X_7} = \frac{(\bar{y}_2+\bar{y}_4+\bar{y}_6+\bar{y}_8)-(\bar{y}_1+\bar{y}_3+\bar{y}_5+\bar{y}_7)}{S_R\sqrt{\sum\frac{1}{n_i}}} \tag{2.39}$$

By comparison of arithmetic values of the t-criterion with tabular ones, we can see that the effects of factors X_1, X_4 and X_7 are statistically significant. Screening then, continues by response correction or by annulling the screened out effects and repeating the procedure. Finally, the following effects were screened out and compared to additional effects in the phase of defining this example.

Factors	Given effects	Estimated effects	Multiple regression effect estimate
X_7	15	13	15.9
X_4	12	12	12.1
$X_{10}X_{11}$	10	8	10.1
X_1	8	11	8.7
X_5X_8	6	5	6.2
X_3	4	4	4.9
X_9	4	5	5.1

Former experience of the author of this book, in applying the method of random balance, indicates that there have been no situations where this method has not

screened out significant factors. Even if a case appears where no most significant factors may be selected, the response variance will not be reduced, even after correcting the response. This means that the most important factors have not been included into the experiment. This, of course, does not mean that it is a method error.

Problem 2.5
The method of prior ranking factors has been unsuccessfully applied on the data in Problem 2.4 about factors that affect the petroleum oils refining procedure by phenol. A design matrix was constructed for this reason and for all sixteen factors, for an experimental screening by the method of random balance. The design matrix with outcomes of the experiment is shown in Table 2.42. Process the results by the method of random balance.

2.2.3 Active Screening Experiment Plackett-Burman Designs

There is annother standard two-level design as active screening design, which the literature recommends, that provides the choice of 4, 8, 16, 32 or more trials-runs, but only the power of two. In 1946, *Plackett and Burman* [64] invented the alternative two-level designs that are multiples of four. The 12-, 20-, and 28-run Plackett-Burrman designs are of particular interest, because they fill gaps in the standard designs. Unfortunately, these particular Plackett-Burman designs have very messy alias structures. For example, the 11th factor in the 12-runs choice, which is very popular, causes each main effect to be partially aliased with 45 two-factor interactions. In theory, you can get away with this if absolutely no interactions are present, but this is a very dangerous assumption. Because of the unexpected aliasing that occurs with many Plackett-Burman designs, it is recommended to avoid them in favor of the standard two-level designs.

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Some of the available software packages for design of experiments use Plackett-Burman designs.

Problem 2.6 [12]
In an optimization process of isomerization of sulfanilamide, a design of experiments has been in the first phase defined by a method of random balance with the idea of doing a screening active experiment. The design of experiments with its results is shown in Table 2.43. Screen factors by significance of their effects on the measured value.

Table 2.42: Design of experiment

	Factors																Responses						
Null level	0.5	1.65	65	90	5	7	25	15	430	I	1.5	5	5	35	35	200							
Interval	0.15	0.6	5	10	2	2	10	5	50	II	1	2	2	10	10	40							
Upper level(+)	0.55	2.25	70	100	7	9	35	20	480	III	2.5	7	7	45	45	240							
Lower level (-)	0.35	1.05	60	80	3	5	15	10	380	II	0.5	3	3	25	25	160							
Number of d.p.	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}	X_{16}	y	y^I	y^{II}	y^{III}	y^{IV}	y^V	$\widehat{y}$
1	+	−	−	+	−	−	+	+	−	−	+	+	−	+	+	+	67.2	45.8	31.2	16.1	5.1	-3.7	79.65
2	+	−	+	−	−	+	−	−	+	−	−	+	−	+	+	−	63.4	42.0	12.9	5.3	-5.7	-25.7	-66.9
3	-	+	−	−	−	+	+	−	−	+	+	+	−	+	−	+	57	46.3	31.7	26.4	15.4	-4.6	66.8
4	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	−	68	46.6	17.5	2.4	-16.2	-36.2	73.09
5	+	+	−	−	+	−	−	−	−	+	−	−	+	+	+	+	89	67.6	53.0	45.4	26.8	18.0	70.55
6	-	+	+	+	−	−	−	+	+	+	−	−	−	+	−	−	87	76.3	47.2	32.1	21.1	12.3	52.05
7	+	−	+	+	+	−	−	−	−	+	+	+	−	−	+	+	90	78.6	49.5	34.4	15.8	7.0	54.4
8	-	−	−	−	+	+	+	+	+	+	−	−	−	−	+	−	42.5	31.8	17.2	9.0	-9.0	-29.0	73.43
9	-	−	+	−	+	−	+	−	+	−	+	−	+	+	−	+	43	32.3	3.2	-4.4	-22.0	-30.3	67.05
10	-	−	−	+	+	+	−	+	−	−	−	+	+	+	−	−	48	38	23.4	8.3	-10.3	-30.8	68.25
11	-	+	+	−	−	−	+	+	−	−	−	+	+	−	+	+	45	34.3	5.2	-2.4	-13.5	-22.3	45.15
12	-	−	+	+	−	+	+	−	−	+	−	−	+	−	−	−	47	36.6	7.2	-7.9	-18.9	-38.9	52.85
13	+	−	−	−	−	−	−	+	+	+	+	+	+	−	−	+	49	27.6	13.0	5.4	-5.6	-14.4	71.75
14	+	+	+	−	+	+	−	+	−	−	+	−	−	−	−	−	86	64.6	35.5	27.9	9.3	-10.7	76.55
15	+	+	−	+	−	+	−	−	+	−	+	−	+	−	+	+	90	68.6	54.0	38.9	27.9	7.9	86.15
16	-	+	−	+	+	−	+	−	+	−	−	+	−	−	−	−	82	71.3	56.7	41.6	23.0	14.2	68.45

Note: $S_y^2 = 73.5$; $S_R^2 = 535$

Table 2.43 Design of experiment

No. D.P.	Factors										Responses	
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	Y	y^I
1	+	+	+	−	−	+	−	−	+	−	67.5	2.02
2	−	+	−	−	−	+	−	+	−	−	83.7	18.20
3	+	−	+	+	−	+	+	+	+	+	27.8	35.31
4	+	−	+	−	+	+	−	−	+	+	21.6	37.40
5	−	−	−	−	+	+	−	+	−	−	5.0	12.51
6	−	+	−	+	−	+	+	−	+	−	84.8	19.32
7	+	+	−	−	−	−	−	−	−	+	67.5	9.75
8	−	−	+	+	−	−	−	+	−	+	8.5	16.85
9	+	−	−	+	+	+	+	−	+	+	9.7	25.56
10	+	+	+	+	+	−	+	+	+	−	70.5	13.30
11	−	−	+	−	+	−	−	−	+	+	7.5	23.36
12	+	−	−	−	+	−	+	−	−	−	7.2	14.71
13	+	+	−	+	−	−	+	−	+	−	70.5	5.02
14	−	+	+	+	+	−	−	+	−	+	85.2	35.58
15	−	+	+	−	+	+	+	+	−	−	84.8	26.83
16	−	−	−	+	−	−	+	+	−	+	8.0	16.35

2.2.3 Completely Randomized Block Design

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In empirical or experimental research it is necessary to determine system stability and reproducibility of the results. In the case of an experiment with a larger number of design points there appears to be a problem of providing the same conditions for doing the design points. The experiment is therefore designed so as to do research in groups of design points *blocks,* where equality of conditions is higher than in the complete field of research. In this way we can single out the effects of inequality of conditions from other factors. The design of experiments that provide this are called *completely randomized block design*. Such designs are used in research with a single factor or they belong to the single-factor design group. Results of an experiment done by a randomized complete block design are analyzed by the method of analysis of variance, which has been elaborated in detail in Sect. 1.5.

Since these randomized blocks are applied to single out inequality effects of a research subject from factor effects, the variance of analysis confidence is increased as experimental error is diminished. The block denotes the part of design points where experimental error is lower than in the experiment as a whole.

To screen out the effects of systematic errors, the effects of factor-level variations are researched in each block by random order. This is the origin of the term randomized complete-block design. These blocks originate from studies in agronomy, for in it there appeared the most drastic case of inequality of agricultural lots where

experiments have been done. To eliminate this inequality, the lots have been divided into blocks or more equal areas. The size and number of blocks primarily depends on the research subject and possibility to equalize experimental conditions, and they are two counter-balanced requirements on which processing confidence of results depends. A small number of blocks means simple calculations of analysis of variance but also greater lack of confidence due to their inequality. When designs of completely randomized blocks are used, an assumption is introduced that outcome levels may be different in different blocks but that relative factor effects are the same in all blocks. This assumption means in practice that there is no interaction between blocks and factors, i.e. even if it exists it is negligible in relation to the factor effect. Interaction is part of experimental error and when it is large, inferences of analysis of variance are not certain. One of the basic objectives of designing experiments by completely randomized blocks, when an experiment is done in full-scale plants, is that the time element is reduced. In a normal operation of a chemical plant, in batches or continuosly, systematic variations in product properties appear. Sometimes these variations may be explained by seasonal influences, such as change in temperature of cooling water, for example, a change in quality of raw materials, etc. However, frequently there are neither explanations for the mentioned fluctuations nor can they can be controlled. The question is not about temporary, random variations that are considered normal in production, but about slow changes in the average level. Therefore, in designing an experiment in such plants, one must take care of separating the effects of temporary trends. This does not mean that the designs of completely randomized blocks are limited only to full-scale. But on the contrary, they are limited to labs and pilot plants where trials for one experiment are done in a longer time period and where there exists a probability of systematic variations and trends [21].

Example 2.14 [21]

In a production plant, it should be determined experimentally whether four kinds of catalyst preparations affect the yield. Namely, four methods of preparing catalysts in a 6-month time period have been researched. One should thereby know that each trial lasts one week. If each catalyst is being researched for a month, there may appear differences due to changes in the efficiency level of the plant, a change in raw materials or the like. If each catalyst is tested for seven days and all four in later weeks, the variation effect is less significant, but the experiment loses its precision, since each catalyst is being researched in a shorter time period. To profit from the six months available for the experiment, it is designed in such a way to split the entire period into six *blocks*, with testing all four catalysts in a months time. To avoid the influence of systematic errors, the catalysts are tested within a month or block in a completely random order. When a linear change in plant efficiency during the experiment is expected in advance, then this inequality of research subject may be eliminated by applying designs of experiments known as *latin squares*.

It should be noted once again that effects of the four catalysts relatively, with respect to each other, remain the same in each month, i.e. there is no interaction between blocks (months) and factor levels (catalyst types). When such an interaction

exists, but it is not great when compared to the factor effect, the experiment will be satisfactory. The sensitivity will be reduced since the experiment error has increased for the effect of interaction.

Analysis of experiment results by design of completely randomized blocks

Since the experimental results, by design of completely randomized blocks, are processed by analysis of variance, experimental results of randomized blocks will be presented as a two-way classification and notation, as introduced in Sect. 1.5. We only introduce the change that the measured values or response are marked by y_{ij} and factors by X_{ij}. Design of completely randomized block structure is given in Table 2.44

Table 2.44 Structure of design of completely randomized blocks

Factor levels	Blocks					Average values
	1	2	3	...	J	
1	Y_{11}	Y_{12}	Y_{13}	...	Y_{1J}	$\bar{Y}_{1\bullet}$
2	Y_{21}	Y_{22}	Y_{23}	...	Y_{2J}	$\bar{Y}_{2\bullet}$
3	Y_{31}	Y_{32}	Y_{33}	...	Y_{3J}	$\bar{Y}_{3\bullet}$
.	.	.	.	...	.	.
I	Y_{I1}	Y_{I2}	Y_{I3}	...	Y_{IJ}	$\bar{Y}_{I\bullet}$
Average values	$\bar{Y}_{\bullet 1}$	$\bar{Y}_{\bullet 2}$	$\bar{Y}_{\bullet 3}$	...	$\bar{Y}_{\bullet J}$	$\bar{Y}_{\bullet\bullet}$

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Table 2.45 Analysis of variance of completely randomized blocks

Source of variations	f	Sum of squares definitions	Sum of squares practical calculations	Mean square	Test statistic
Between columns of blocks	J-1	$SS_C = I\sum_j \left(\bar{Y}_{\bullet j} - \bar{Y}_{\bullet\bullet}\right)^2$	$SS_C = \frac{\sum_j Y_{\bullet J}^2}{I} - \frac{Y_{\bullet\bullet}^2}{IJ}$	$MS_C = \frac{SS_C}{J-1}$	$\frac{MS_C}{MS_E}$
Between rows of factor levels	I-1	$SS_R = J\sum_i \left(Y_{i\bullet} - \bar{Y}_{\bullet\bullet}\right)^2$	$SS_R = \frac{\sum_i Y_{i\bullet}^2}{J} - \frac{Y_{\bullet\bullet}^2}{IJ}$	$MS_R = \frac{SS_R}{I-1}$	$\frac{MS_R}{MS_E}$
Residual variance of error	(J-1)(I-1)	$SS_E = \sum_i\sum_j \left(Y_{ij} - \bar{Y}_{i\bullet} - \bar{Y}_{\bullet j} + \bar{Y}_{\bullet\bullet}\right)^2$	$SS_E = SS_T - SS_C - SS_R$	$MS_E = \frac{SS_E}{(I-1)(J-1)}$	–
Total	IJ-1	$SS_T = \sum_i\sum_j \left(Y_{ij} - \bar{Y}_{\bullet\bullet}\right)^2$	$SS_T = \sum_i\sum_j Y_{ij}^2 - \frac{Y_{\bullet\bullet}^2}{IJ}$	–	–

The experiment is done by the design shown in Table 2.44, and the obtained results processed by analysis of variance. Definitions with calculation forms of analysis of variance are shown in Table 2.45.

Although block effects are less interesting for the research objective, the large variance value between the blocks means that division into blocks was justified, i.e. that residual variance has been reduced, which contributed to a higher precision of the experiment. A high variance value between the blocks means at the same time that one should find out the causes for such an expressed inequality of experiment conditions. It should be noted that analysis of variance in Table 2.45 comprises performing the experiment by design of randomized blocks without repeating the measurements.

The basic assumption of this kind of design of experiments without repeating measurements is that each measured result may be described by this model.

$$Y_{ij} = \mu + \alpha_i + \beta_i + \varepsilon_{ij} \qquad (2.40)$$
$$i=1, 2,..., I; \quad j=1, 2,..., J$$

where:
μ is actual mean effect;
β_j is actual block effect;
α_i is actual factor effect;
ε_{ij} is experimental error with normal distribution $N(O,\sigma^2)$.

Experiments may be done by design of completely randomized blocks and by repeating measurements in which case analysis of variance has a different form (Table 2.46):

It should be noted that the number of measurement replications in the matrix of design of completely randomized blocks is marked by K. A distinction should also be made between mean squares for measurement error + experimental error and measurement error. Often this sum of measurement and experimental errors is just called *experimental error*, and measurement error *sampling error*. To check significance of the factor effect, the mean square of joint error or experimental error MS_{CR} is used.

Example 2.15 [21]

Studies of chlorosulphonizing of acetaniline showed that the obtained yield of chemical reaction was considerably below theoretical due to losses in the mother liquid. Research of the effects of five possible mixtures of acetaniline on yield was suggested. These five mixtures were marked as A, B, C, D and E. Experiments lasted for 15 days, and since production of one batch of acetaniline lasts 24h, the whole experimental program was divided into three blocks of five batches each. The outcomes are shown in Table 2.47. The results were processed by analysis of variance and shown in Table 2.48. These results at 95% confidence coefficient were neither considerable between blocks nor was the effect of different acetaniline mixtures significant. However, at 90% confidence $F_{4;8;0.90}=2.81$, so that the effect of different acetaniline mixtures was statistically significant. Such a high level of risk in passing a decision demands further research to select the best option of technological procedure

Table 2.46 Analysis of variance of completely randomized blocks with replication

Source of variations	f	Sum of squares definitions	Sum of squares practical calculations	Mean square	Test statistic
Between blocks	J-1	$IK\sum_j \left(\bar{Y}_{\bullet j\bullet} - \bar{Y}_{\bullet\bullet\bullet}\right)^2$	$SS_C = \frac{\sum_j Y^2_{\bullet j\bullet}}{IK} - \frac{Y^2_{\bullet\bullet\bullet}}{IJK}$	$MS_C = \frac{SS_C}{J-1}$	–
Between factor levels	I-1	$JK\sum_i \left(\bar{Y}_{i\bullet\bullet} - \bar{Y}_{\bullet\bullet\bullet}\right)^2$	$SS_R = \frac{\sum_i Y^2_{i\bullet\bullet}}{JK} - \frac{Y^2_{\bullet\bullet\bullet}}{IJK}$	$MS_R = \frac{SS_R}{I-1}$	$F = \frac{MS_R}{MS_{CR}}$
Measurement error + experimental error	(J-1)(I-1)	$K\sum_i\sum_j \left(\bar{Y}_{ij\bullet} - \bar{Y}_{i\bullet\bullet} - \bar{Y}_{\bullet j\bullet} + \bar{Y}_{\bullet\bullet\bullet}\right)^2$	$SS_{CR} = \frac{\sum_i\sum_j Y^2_{ij\bullet}}{K} - \frac{\sum_i Y^2_{i\bullet\bullet}}{JK} - \frac{\sum_j Y^2_{\bullet j\bullet}}{IK} + \frac{Y^2_{\bullet\bullet\bullet}}{IJK}$	$MS_{CR} = \frac{SS_{CR}}{(J-1)(I-1)}$	–
Measurement error	IJ(K-1)	$\sum_i\sum_j\sum_k \left(Y_{ijk} - \bar{Y}_{i\bullet\bullet} - \bar{Y}_{\bullet j\bullet} + \bar{Y}_{\bullet\bullet\bullet}\right)^2$	$SS_E = \sum_i\sum_j\sum_k Y^2_{ijk} - \frac{\sum_i\sum_j Y^2_{ij\bullet}}{K}$	$MS_E = \frac{SS_E}{IJ(K-1)}$	–
Total	IJK-1	$\sum_i\sum_j\sum_k \left(Y_{ijk} - \bar{Y}_{\bullet\bullet\bullet}\right)^2$	$\sum_i\sum_j\sum_k Y^2_{ijk} - \frac{Y^2_{\bullet\bullet\bullet}}{IJK}$	–	–

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for acetaniline production. Analysis of variance showed that there were no significant differences between blocks, i.e. that no trend of changes in levels of obtained results was noticed.

Table 2.47 Design of experiment

Block	Batch	Acetaniline mixture	Percentage of loss
I	1	B	18.2
	2	A	16.9
	3	C	17.0
	4	E	18.3
	5	D	15.1
II	6	A	16.5
	7	E	18.3
	8	B	19.2
	9	C	18.1
	10	D	16.0
III	11	B	17.1
	12	D	17.8
	13	C	17.3
	14	E	19.8
	15	A	17.5

Table 2.48 Analysis of variance

Source of variation	f	SS	MS	F	$F_{2;8;0.95}$
Blocks	2	1.65	0.82	0.94	4.46
Factor levels	4	11.56	2.89	3.32	3.84
Error	8	6.99	0.87	–	–
Total	14	20.20	–	–	–

Example 2.16 [22]

In an experiment designed as completely randomized blocks, the effect of Co % on steel tensile strength was researched. Three vessels for producing alloys were used in experimental procedure. Each measurement of tensile strength was repeated and outcomes are shown in thousands of PSI-a in Table 2.49.

Table 2.49 Completely randomized blocks with measurements replications

Vessel-block	1% Co		2% Co		3% Co		4% Co	
1	49	50	60	62	64	67	71	75
2	44	45	53	56	63	65	65	67
3	53	56	64	65	74	78	76	80

The results of analysis of variance are shown in Table 2.50.

Table 2.50 Analysis of variance

Sources of variations	f	SS	MS	F	$F_{f;6;0.95}$
Blocks	2	485.3	242.65	42.80	5.14
Factor levels	3	1847.5	615.83	108.61	4.76
Experimental error	6	34.0	5.67	–	–
Sampling error	12	45.0	3.75	–	–
Total	23	2411.8	–	–	–

Problem 2.7 [22]

In the previous experiment, where the effect of cobalt contents on steel tensile strength was researched, we are now trying to find out the effect of three percentile contents of cobalt on this response if each alloy was tempered in each of four furnaces. Twelve alloy batches were divided into three samples each, so that the tensile strength for 36 samples were measured. These values in thousands of PSI for measurements or determinations are shown in Table 2.51. Do the analysis of variance.

Table 2.51 Completely randomized blocks with measurement replications

OK	1% Co			2% Co			3% Co		
1	50	47	55	64	59	55	60	66	69
2	43	42	51	48	55	62	59	60	70
3	49	53	57	66	63	65	67	83	75
4	45	49	55	67	63	60	63	72	70

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Problem 2.8

Process the results of previous problem by analysis of variance assuming that an experiment by design of completely randomized blocks was done with no measurement replications. Use the means of replicated measurements for such an analysis, as shown in Table 2.52

Compare the results of analysis from the previous and from this problem.

Table 2.52 Completely randomized blocks with no replications

Furnace block	1% Co	2% Co	3% Co
1	50.67	59.33	65.00
2	45.33	55.00	63.00
3	53.00	64.67	75.00
4	49.67	63.33	68.33

2.2.3.1 Incomplete Random Block Design

Former analysis of designs of completely randomized blocks included designs for all combinations of factor and block variation levels. In doing these designs in practice certain results may be lacking due to the impossibility of measurement, lack of material, error in measurement, etc. According to the number and position of the missing data one can distinguish *balanced* and *unbalanced* incomplete random blocks. Since balanced are a special case of unbalanced incomplete random blocks, only unbalanced designs will be analyzed [23]. Let us analyze (I×J)-dimensional order of experimental results with K missing values, Table 2.53.

Table 2.53 Incomplete random blocks

Factor levels	Block				
	1	**2**	**3**	**...**	**J**
1	Y	–	Y	...	–
2	–	Y	Y	...	Y
3	Y	Y	–	...	Y
.	.	.	.	.	.
I	Y	Y	–	...	Y

Estimate of values is done in order to complete the analysis of variance even when some values are lacking. There are two methods for this estimate:

- direct method, when fewer data than the sum of lines and columns are missing;
- constant method, when more data than the sum of lines and columns are missing.

Direct methods

Missing data Z_{ij} are determined from the condition that residual sum of squares SS_E is minimal.

$$SS_E = SS_T - SS_C - SS_R \tag{2.41}$$

where:

SS_T are sums of squares of deviations of individual results from the mean of all results;

SS_C are sums of squares of deviations of block centers from the mean of all results;

SS_R are sums of squares of deviations of factor centers from the mean of all results.

If we now mark the sum of all individual values by S

$$S = \sum_i \sum_j \left(Y_{ij} + Z_{ij} \right)$$

the sums of squares may be written as:

$$SS_T = \sum\sum Y_{ij}^2 + \sum\sum Z_{ij}^2 - \frac{S^2}{IJ} \tag{2.42}$$

$$SS_C = \frac{\sum_j Y_{\bullet j}^2}{I} - \frac{S^2}{IJ} \tag{2.43}$$

$$SS_R = \frac{\sum_i Y_{i\bullet}^2}{J} - \frac{S^2}{IJ} \tag{2.44}$$

After replacing relations (2.42)–(2.44) in Eq. (2.41) we get:

$$SS_E = \sum_i \sum_j Y_{ij}^2 + \sum_i \sum_j Z_{ij}^2 - \frac{\sum_j Y_{\bullet j}^2}{I} - \frac{\sum_i Y_{i\bullet}^2}{J} + \frac{S^2}{IJ} \tag{2.45}$$

By partial differentiation of residual sum of squares by Z_{ij} and bringing it down to zero, we obtain normal equations. By solving the simultaneous equations we determine individual values of results that are not available.

Example 2.17

When testing durability or wear-out of car tires, the effect of four kinds of plasticizers was researched. Due to problems in doing the experiment, the following outcomes were obtained:

Table 2.54 Incomplete random blocks

Plasticizer Type	Car tire block		
	1	2	3
A	238	196	254
B	238	213	–
C	279	–	334
D	–	308	367

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The values Z_{41}; Z_{32} and Z_{23} are evidently missing, so that we get Table 2.54:

Table 2.55 Incomplete random blocks

Plasticizer type	Car tire			Sum
	1	2	3	
A	238	196	254	688
B	238	213	Z_{23}	$451+Z_{23}$
C	279	Z_{32}	334	$613+Z_{32}$
D	Z_{41}	308	367	$675+Z_{41}$
Sum	$755+Z_{41}$	$717+Z_{32}$	$955+Z_{23}$	$2427+Z_{23}+Z_{32}+Z_{41}$

The residual sum of squares is:

$$SS_E = Z_{41}^2 + Z_{32}^2 + Z_{23}^2 + \sum\sum Y_{ij}^2 - \frac{(451+Z_{23})^2}{3} - \frac{(613+Z_{32})^2}{3} - \frac{(675+Z_{41})^2}{3} - \frac{(755+Z_{41})^2}{4} - \frac{(717+Z_{32})^2}{4} - \frac{(955+Z_{23})^2}{4} + \frac{(2427+Z_{23}+Z_{32}+Z_{41})^2}{12}$$

By partial differentiation SS_E/Z_{23}; SS_E/Z_{32}; SS_E/Z_{41} and bringing it down to zero we have:

$$\begin{cases} 6Z_{41} + Z_{32} + Z_{23} = 2538 \\ Z_{41} + 6Z_{32} + Z_{23} = 2176 \\ Z_{41} + Z_{32} + 6Z_{23} = 2242 \end{cases} \tag{2.46}$$

By calculating the system of linear equations we get:

Z_{41}=333.7; Z_{32}=261.3; Z_{23}=274.5.

Constant method

Let us mark block effects by b_1, b_2, ..., b_J, factor effects by a_1, a_2, ..., a_I , and by m the mean of all results. By definition $\sum b_j = \sum a_i = 0$, and the number of independent constants for estimates is equal to: number of rows + number of columns -1. Let Y_{ij} denote the response value in row i and column j. The expected value in i rows and j columns based on columns effects and rows effects is $a_i + b_j + m$, while the deviation of experimental values Y_{ij} from it is:

$$Y_{ij}-(a_i+b_j+m) \tag{2.47}$$

The values that are not available are estimated from the condition that the deviation sum of squares is minimal.

$$S = \sum \left[Y_{ij} - \left(a_i + b_j + m \right) \right]^2 \tag{2.48}$$

Values a_i, b_j and m are obtained from this system of normal equations:

$$\begin{cases} \frac{1}{2}\frac{\partial S}{\partial m} = \sum\sum \left(Y_{ij} - m - b_j - a_i \right) = 0 \\ \frac{1}{2}\frac{\partial S}{\partial b_1} = \sum \left(Y_{i1} - m - b_1 - a_i \right) = 0 \\ \cdots \qquad\qquad \cdots \\ \frac{1}{2}\frac{\partial S}{\partial b_J} = \sum \left(Y_{iJ} - m - b_J - a_i \right) = 0 \\ \frac{1}{2}\frac{\partial S}{\partial a_1} = \sum \left(Y_{1j} - m - b_j - a_1 \right) = 0 \\ \cdots \qquad\qquad \cdots \\ \frac{1}{2}\frac{\partial S}{\partial a_I} = \sum \left(Y_{Ij} - m - b_j - a_I \right) = 0 \end{cases} \tag{2.49}$$

Example 2.18

Expected values of experimental results are shown in Table 2.56 (Example 2.17).

Deviation sum of squares is obtained by squaring the difference of expected values (Table 2.56) and experimentally obtained results (Table 2.54). By partial differentiation of a_i, b_j and m and bringing it down to zero, we get the following system of normal equations:

Table 2.56 Incomplete random blocks

Factor levels	Block		
	1	2	3
1	$m+a_1+b_1$	$m+a_1+b_2$	$m+a_1+b_3$
2	$m+a_2+b_1$	$m+a_2+b_2$	–
3	$m+a_3+b_1$	–	$m+a_3+b_3$
4	–	$m+a_4+b_2$	$m+a_4+b_3$

$$\begin{cases} 2427 - 9m - 3(b_1 + b_2 + b_3) - 3a_1 - 2(a_2 + a_3 + a_4) = 0 \\ 688 - 3m - 3a_1 - (b_1 + b_2 + b_3) = 0 \\ 451 - 2m - 2a_2 - (b_1 + b_2) = 0 \\ 613 - 2m - 2a_3 - (b_1 + b_3) = 0 \\ 675 - 2m - 2a_4 - (b_2 + b_3) = 0 \\ 755 - 3m - 3b_1 - (a_1 + a_2 + a_3) = 0 \\ 717 - 3m - 3b_2 - (a_1 + a_2 + a_4) = 0 \\ 955 - 3m - 3b_3 - (a_1 + a_3 + a_4) = 0 \end{cases} \quad (2.50)$$

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Calculations of simultaneous system of equations are:

$m=274.708$; $b_1=-2.533$; $b_2=-30.133$; $b_3=32.667$; $a_1=-45.375$; $a_2=-32.875$; $a_3=16.725$; $a_4=61.525$

The values that are not available are determined from obtained constants through Equ. (2.47):

$Y_{ij}=m+a_i+b_j$

For example for Z_{41} we get the same as for the direct method:

$Z_{41}=m+a_4+b_1=274.708+61.525-2.533=333.7$

Although this direct method is more adequate for the given example, because the number of the values that are not available are smaller than the sum of rows and columns, the constant method has also been demonstrated for the case of comparison. It should be noted that both methods are generally used in two-way classification such as designs of completely randomized blocks, Latin squares, factorial experiments, etc. Once the values that are not available are estimated, the averages of individual blocks and factor levels are calculated and calculations by analysis of variance done. The degree of freedom is thereby counted only with respect to the number of experimental values. Results of analysis of variance for this example are

shown in Table 2.57. The total number of degrees of freedom is 8 and not 3×4-1=11, since three values have been estimated. Residual variance in incomplete random blocks therefore has a higher value than in complete ones, so the F-test is less sensitive.

Table 2.57 Analysis of variance of incomplete random blocks

Sources of variation	f	SS	MS	F	$F_{f;3;0.95}$
Blocks	2	4933.87	2466.94	17.36	9.55
Factor levels	3	14733.03	4911.01	36.56	9.28
Error	3	426.30	142.10	–	–
Total	8	20093.20	–	–	–

It is clear from the table of analysis of variance that the factor effect is statistically highly significant. The effect of blocks is also important, which justifies the division of experimental conditions into blocks.

2.2.4 Latin Squares

Designs of experiments that are specially useful in research, development and optimization in the phase of screening factors are called *Latin squares*. As for randomized blocks that are used to eliminate one cause of inequality (nonhomogeneity) of a research subject, Latin squares are applied to distinguish two causes of inequality of a research. Inequality of experimental conditions is reduced even more by applying Latin squares, which facilitates a more precise analysis of the effect of researched factor. Since Latin squares are primarily used in single-factorial experiments for researching the effect of one factor when double inequality or double division into randomized blocks of a research subject is present, one may say that Latin squares are an expansion of designs of completely randomized blocks or 1/m replica of type m^3 full factorial experiment.

Example 2.19

Consider an experiment where durability or wear-out of four types of car tires have to be researched. Sixteen tires are at our disposal, four of each type. The research will be done on four cars. The factor in this case is the type of car tire. There are, however, two additional factors that affect the durability of tires:

- car type,
- tire position on car.

Let us mark car type as I, II, III and IV and the position of tires on each car as FR, FL, RL and RR. The latest two factors are singled out as an inequality of experimental conditions into blocks by rows and columns. Car tire types are placed by random choice on cars but so that one type of tire is put on one type of car only once in the same position.

Design of Latin squares is frequently applied when the effect of one factor on several conditionally the same devices is researched for a long time. In that case, rows of designs correspond to successive time studies, and columns to experimental devices.

Experimental designs are square in forms (m×m), and the researched factor is tested once in each step. Table 2.58 shows an example of 4×4 Latin square design.

Table 2.58 Design of experiment of latin square

Rows	Columns			
	1	2	3	4
1	A	B	C	D
2	B	C	D	A
3	C	D	A	B
4	D	A	B	C

It is clear from the table that A, B, C and D are levels of the researched factor. An important condition for applying design of Latin squares is that in each column and each row one factor level may appear once and only once.

Analysis of variance of latin squares

The model for a general m x m Latin square with one observation in the cell is:

$$Y_{ij(k)} = \mu + \alpha_i + \beta_j + \tau_k + \varepsilon_{ij(k)} \\ = 1.2, \ldots, m; j = 1.2, \ldots, m; k = 1.2, \ldots, m. \quad (2.51)$$

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where:

$$\sum \alpha_i = \sum \beta_j = \sum \tau_k = 0$$

$\varepsilon_{ij(k)}$ has normal distributions $N(0, \sigma^2)$.

Variables α_i, β_j and τ_k are actual effects of i rows, j columns and the k factor level.

One can notice that the k index is bracketed to indicate that in the design of Latin squares there are no m results, as is the case with a three-factorial design with one design-point replication. Design of Latin squares actually has m^2 observations or data.

Analysis of variance for an m x m Latin square with one observation per cell in concordance with model (2.51) is shown in Table 2.59. Associated sums of data per rows, columns and factor are marked $Y_{i\bullet(\bullet)}$, $Y_{\bullet j(\bullet)}$ and $Y_{\bullet\bullet(k)}$.

Table 2.59 shows that total variance of experimental results is divided into variances of:

- R row-variation sources;
- C column-variation sources;
- T factor-variation sources;
- residual or experimental error.

The condition for application of Latin squares are interactions that are negligible with respect to experimental error. As in researching complex system there exist interactions, Latin square designs are not widely applied.

Table 2.59 Analysis of variance of m × m latin square

Source of variation	f	SS	MS
Rows	m-1	$SS_R = \frac{\sum_i Y^2_{i\bullet(\bullet)}}{m} - \frac{Y^2_{\bullet\bullet(\bullet)}}{m^2}$	$MS_R = \frac{SS_R}{m-1}$
Columns	m-1	$SS_C = \frac{\sum_j Y^2_{\bullet j(\bullet)}}{m} - \frac{Y^2_{\bullet\bullet(\bullet)}}{m^2}$	$MS_C = \frac{SS_C}{m-1}$
Factor	m-1	$SS_T = \frac{\sum_k Y^2_{\bullet\bullet(k)}}{m} - \frac{Y^2_{\bullet\bullet(\bullet)}}{m^2}$	$MS_T = \frac{SS_T}{m-1}$
Residual	(m-1)(m-2)	$SS_E = \sum_i \sum_j \sum_k Y^2_{ij(k)} - \frac{Y^2_{\bullet\bullet(\bullet)}}{m^2} - SS_R - SS_C - SS_T$	$MS_E = \frac{SS_E}{(m-1)(m-2)}$
Total	m^2	$\sum_i \sum_j \sum_k Y^2_{ij(k)} - \frac{Y^2_{\bullet\bullet(\bullet)}}{m^2}$	–

Example 2.20 [15]

As already defined in Example 2.19, four types of tires A, B, C and D have been researched by design of Latin squares in this way: sixteen tires, four of each type, have been put on four cars (I, II, III and IV) under these conditions:

- each car has one tire of each of the four types;
- each type of tire is in positions FL, FR, RL and RR;
- actual position of each of 16 tires has been randomly chosen for 4 × 4 Latin square.

Wear-out of tires has been measured after 8000 km by a standard procedure. The obtained results are shown in Table 2.60.

Table 2.60 Design of experiment-latin square

Car	Position				$Y_{i\bullet(\bullet)}$
	FR	FL	RR	RL	
I	A 31	B 33	C 47	D 54	165
II	B 36	D 53	A 42	C 54	185
III	C 51	A 43	D 62	B 49	205
IV	D 81	C 78	B 72	A 84	315
$Y_{\bullet j(\bullet)}$	199	207	223	241	870

After calculation we obtain:

$$\frac{\sum Y^2_{\bullet\bullet(\bullet)}}{16} = \frac{870^2}{16} = 47306.25;$$

$$\frac{\sum\limits_j Y^2_{\bullet j(\bullet)}}{4} = \frac{199^2+207^2+223^2+241^2}{4} = \frac{190260}{4} = 47565;$$

$$\frac{\sum\limits_i Y^2_{i\bullet(\bullet)}}{4} = \frac{165^2+185^2+205^2+315^2}{4} = \frac{202700}{4} = 50675;$$

$$\frac{\sum\limits_k Y^2_{\bullet\bullet(k)}}{4} = \frac{(31+43+42+84)^2+\ldots+(81+53+62+54)^2}{4}$$

$$= \frac{200^2+190^2+230^2+250^2}{4} = 47875.$$

So that:

$$SS_R = \frac{\sum\limits_i Y^2_{i\bullet(\bullet)}}{4} - \frac{Y^2_{\bullet\bullet(\bullet)}}{16} = 3368.75;$$

$$SS_C = \frac{\sum\limits_j Y^2_{\bullet j(\bullet)}}{4} - \frac{Y^2_{\bullet\bullet(\bullet)}}{16} = 258.75;$$

$$SS_T = \frac{\sum\limits_k Y^2_{\bullet\bullet(k)}}{4} - \frac{Y^2_{\bullet\bullet(\bullet)}}{16} = 568.75;$$

$$SS_E = \sum\sum\sum Y^2_{ij(k)} - \frac{Y^2_{\bullet\bullet(\bullet)}}{16} - SS_R - SS_C - SS_T$$

$$= 51540.00\text{-}47306.25\text{-}3368.745\text{-}258.75\text{-}568.75\text{=}37.50.$$

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Table 2.61 Analysis of variance

Sources of variation	f	SS	MS	F	$F_{f;6;0.95}$
Rows cars	3	3368.75	1122.917	179.66	4.76
Column positions	3	258.75	86.250	13.80	4.76
Factor tire type	3	568.75	189.583	30.33	4.76
Experimental error	6	37.50	6.250	–	–
Total	15	4233.75	–	–	–

Statistically significant differences between car tires, tire positions and tire types are clearly evident from analysis of variance. Table 2.60 indicates the highest wear out of tires on car IV and the lowest on car I. This does not present an error but is simply the upper and lower limit of tire wear-out in this research.

Example 2.21 [24]

In finding the composition for neutral light filters based on poly methyl methacrylate (PMMA) it was necessary to research the effect of four different dyestuffs (X_3), which are prepared in four different ways (X_2) and in four concentrations (X_1) as for the filter optical properties. The experiment was done by a 4 x 4 design of Latin square. Samples of obtained PMMA were tested for light permeability within ranges of these spectra: 240–300(Y_1); 300–340(Y_2); 300–1000(Y_3) and for general light permeability (Y_4). *GOST*s demand for light filters is that Y_1 does not exceed 0.5%; Y_2-20%; Y_3-45%, and general permeability to be between 20% and 26%. Since no analyzed response has a universal property, overall desirability D should be taken as a representative estimate of the quality of light filter.

$$D = \sqrt[4]{d_1 \times d_2 \times d_3 \times d_4} \tag{2.52}$$

where

d_i are partial desirabilities.

Desirability scale for individual properties is shown in Figs. 2.26 and 2.27:

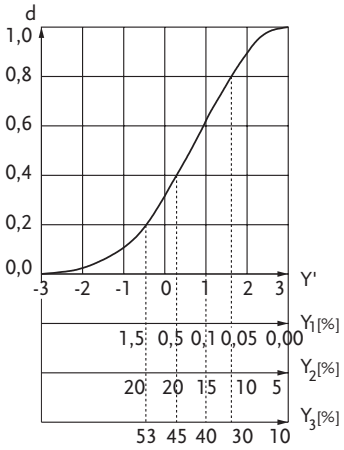

Figure 2.26 Desirability scale

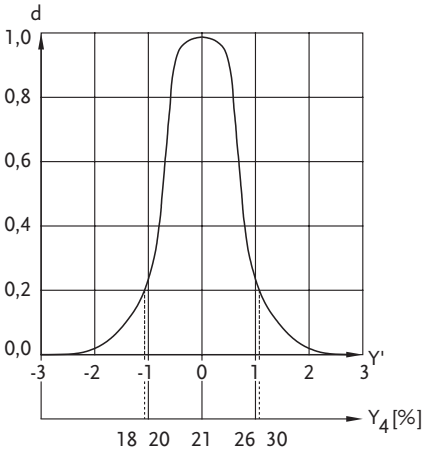

Figure 2.27 Desirability scale

The results of design and performance of experiment by 4x4 Latin square are shown in Table 2.62.

Table 2.62 Design of experiment by 4x4 latin square

Levels of variation	Factors			Responses				
	X_1	X_2	X_3	Y_1	Y_2	Y_3	Y_4	D
0	0.006	1	A					
1	0.008	2	B					
2	0.010	3	C					
3	0.020	4	E					
1	0	0	A	0.1	10.9	35.8	20.5	0.73
2	0	1	B	0.0	46.1	70.1	69.5	0
3	0	2	C	0.1	19.8	72.8	39.5	0
4	0	3	E	0.2	53.0	69.2	66.3	0
5	1	0	B	0.0	44.0	63.8	61.5	0
6	1	1	C	0.0	24.0	72.8	40.9	0
7	1	2	E	0.0	22.5	65.3	48.8	0
8	1	3	A	0.0	5.0	20.1	22.1	0.83
9	2	0	C	0.0	21.0	70.3	34.0	0
10	2	1	E	0.1	32.8	53.8	51.0	0
11	2	2	A	0.0	3.0	29.8	12.8	0.50
12	2	3	B	0.0	16.5	50.8	35.0	0.34
13	3	0	E	0.0	10.0	37.0	31.9	0.66
14	3	1	A	0.1	0.5	9.0	2.3	0
15	3	2	B	0.1	18.0	36.0	26.3	0.67
16	3	3	C	0.0	7.0	70.8	25.8	0

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The results of analysis of variance are shown in Table 2.63.

Table 2.63 Analysis of variance

Sources of variations	f	SS	MS	F	$F_{f;6;0.95}$
Factor x1	3	0.333042	0.111014	1.77	4.76
Factor x2	3	0.069641	0.023214	0.37	4.76
Factor x3	3	1.573387	0.524462	8.39	4.76
Experimental error	6	0.374770	0.062462	–	–
Total	15	2.350840	–	–	–

Analysis of variance shows that only factor effect X_3 is significant. This means that out of four different dyestuffs one should choose the most convenient one.

■ **Problem 2.9 [21]**

The procedure of spraying an insecticide over a treated area is very important for its efficient use. A study has therefore been done with the idea of establishing the best procedure for preparing a spraying insecticide. The following three factors have been researched:

1. type of component mixing;
2. form of active matter;
3. solvent type.

Under the assumption that there is no significant interaction between the given factors, the design of Latin squares has been applied. In this case, seven solvent types, methods of mixing and forms of active matter have been researched. Stability of insecticide is measured as the response. The results are shown in Table 2.64. Do the analysis of variance.

Table 2.64 Design matrix of 7 × 7 latin square

Type of mixing	Form of active matter							Sum
	1	2	3	4	5	6	7	
1	A 98	B 117	C 89	D 64	E 63	F 123	G 244	807
2	B 69	E 67	A 70	G 70	F 111	D 60	C 218	665
3	C 37	F 83	G 83	B 74	D 70	A 75	E 169	591
4	D 65	G 60	E 91	F 56	C 61	B 59	A 150	542
5	E 56	D 44	B 70	C 68	A 88	G 111	F 220	657
6	F 113	C 105	D 65	A 51	G 83	E 57	B 233	707
7	G 64	A 62	F 65	E 86	B 45	C 108	D 187	617
Sum	502	538	533	469	521	602	1421	4586

■ **Problem 2.10 [15]**

The design of a 4 × 4 Latin square has been used in researching effects of water pressure, air flow and number of nozzles in operation on scrubber efficiency. Research outcomes are shown in Table 2.65. Do the analysis of variance for the given data.

Table 2.65 Design matrix of 4 × 4 latin square

Nozzles in operation	Air flow				Sum
	5120	2560	4160	6400	
N_A	90	81	95	95	361
N_C	–	80	88	85	253
N_D	83	83	88	67	321
N_B	96	95	88	94	373
Sum	269	339	359	341	1308

Problem 2.11 [25]

An alternator for a missile has been constructed and made. The alternator is activated by a turbine, and the turbine by the products of burning of the generator gas from solid rocket propellant. The alternator is composed of three separate sections that generate energy, each of them being independent. One section provides energy for keeping the alternator velocity at 24000 min^{-1}. It is composed of a 4-pole stator, 6-pole rotor and axis. The rotor rotates concentrically in the stator opening, while the stator is fixed in its box. The stator consists of coils for direct and alternating currents. The exit voltage of the alternating current is a function of the inlet direct current and the number of alternating coils. The rotor is coiled by laminates 0.004 inches thick. The laminates are covered up for insulation. The experiment is aimed at finding factors and their levels that affect the alternator performance most significantlly. The design of experiment was a 55 Latin square with these factors and levels:

- number of coils for alternating current of stator are these: 145; 150; 155; 160 and 165;
- number of rotor laminates: 230; 240; 250; 260 and 270;
- visual quality of laminate insulation: A; B; C; D and E, where A is the best and E the worst quality.

The result of measurement is the maximal parasitic voltage of alternating current. All the measured values have been reduced by 300 to make calculation easier and are shown in Table 2.66. Do the analysis of variance.

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Table 2.66 Design of experiment of 5 × 5 latin square

Rotors	Stators					Sum
	145	150	155	166	165	
230	C 10	B 12	A 20	D 6	E 0	48
240	D 9	C 10	B 24	E 0	A 5	48
250	B 12	E 3	C 25	A 7	D 2	49
260	A 16	D 6	E 18	C 4	B -6	38
270	E 14	A 8	D 23	B 9	C 3	57
Sum	61	39	110	26	4	240

The following Latin squares may be used for practical solving of the problem:

4×4

1.

A	B	C	D
B	A	D	C
C	D	B	A
D	C	A	B

2.

A	B	C	D
B	C	D	A
C	D	A	B
D	A	B	C

3.

A	B	C	D
B	D	A	C
C	A	D	B
D	C	B	A

4.

A	B	C	D
B	A	D	C
C	D	A	B
D	C	B	A

5×5

A	B	C	D	E
B	A	E	C	D
C	D	A	E	B
D	E	B	A	C
E	C	D	B	A

6×6

A	B	C	D	E	F
B	F	D	C	A	E
C	D	E	F	B	A
D	A	F	E	C	B
E	C	A	B	F	D
F	E	B	A	D	C

7×7

A	B	C	D	E	F	G
B	C	D	E	F	G	A
C	D	E	F	G	A	B
D	E	F	G	A	B	C
E	F	G	A	B	C	D
F	G	A	B	C	D	E
G	A	B	C	D	E	F

8×8

A	B	C	D	E	F	G	H
B	C	D	E	F	G	H	A
C	D	E	F	G	H	A	B
D	E	F	G	H	A	B	C
E	F	G	H	A	B	C	D
F	G	H	A	B	C	D	E
G	H	A	B	C	D	E	F
H	A	B	C	D	E	F	G

9×9

A	B	C	D	E	F	G	H	I
B	C	D	E	F	G	H	I	A
C	D	E	F	G	H	I	A	B
D	E	F	G	H	I	A	B	C
E	F	G	H	I	A	B	C	D
F	G	H	I	A	B	C	D	E
G	H	I	A	B	C	D	E	F
H	I	A	B	C	D	E	F	G
I	A	B	C	D	E	F	G	H

10×10

A	B	C	D	E	F	G	H	I	J
B	C	D	E	F	G	H	I	J	A
C	D	E	F	G	H	I	J	A	B
D	E	F	G	H	I	J	A	B	C
E	F	G	H	I	J	A	B	C	D
F	G	H	I	J	A	B	C	D	E
G	H	I	J	A	B	C	D	E	F
H	I	J	A	B	C	D	E	F	G
I	J	A	B	C	D	E	F	G	H
J	A	B	C	D	E	F	G	H	I

11 × 11

A	B	C	D	E	F	G	H	I	J	K
B	C	D	E	F	G	H	I	J	K	A
C	D	E	F	G	H	I	J	K	A	B
D	E	F	G	H	I	J	K	A	B	C
E	F	G	H	I	J	K	A	B	C	D
F	G	H	I	J	K	A	B	C	D	E
G	H	I	J	K	A	B	C	D	E	F
H	I	J	K	A	B	C	D	E	F	G
I	J	K	A	B	C	D	E	F	G	H
J	K	A	B	C	D	E	F	G	H	I
K	A	B	C	D	E	F	G	H	I	J

12 × 12

A	B	C	D	E	F	G	H	I	J	K	L
B	C	D	E	F	G	H	I	J	K	L	A
C	D	E	F	G	H	I	J	K	L	A	B
D	E	F	G	H	I	J	K	L	A	B	C
E	F	G	H	I	J	K	L	A	B	C	D
F	G	H	I	J	K	L	A	B	C	D	E
G	H	I	J	K	L	A	B	C	D	E	F
H	I	J	K	L	A	B	C	D	E	F	G
I	J	K	L	A	B	C	D	E	F	G	H
J	K	L	A	B	C	D	E	F	G	H	I
K	L	A	B	C	D	E	F	G	H	I	J
L	A	B	C	D	E	F	G	H	I	J	K

2.2.5
Graeco-Latin Square

By applying Latin squares one could do studies with three independent factors simultaneously or one could analyze the effect of one factor while singling out two inequalities of a research subject, such as for instance, more experimental devices and different times of doing the experiment. The concept of Latin squares may easily be extended to *Graeco-Latin squares*. This kind of design allows the same number of levels of a factor to be experimentally researched on the same number of experimental devices in different times and different locations. Use of these designs results in exceptional savings of time and means, as the total number of design points has been drastically reduced. However, similar to Latin squares, these designs may be used only when interactions are statistically insignificant. Graeco-Latin squares are constructed so that one design of a Latin square with Latin characters is put over another design of a Latin square but with Greek characters, and in such a way that each Graeco-Latin pair of characters appears once and only once. In such conditions one may construct n × n Graeco-Latin squares n≠2,6,10. In constructing Graeco-Latin squares, numbers are frequently used instead of Greek characters. Generally speaking, rows represent levels of one factor, columns levels of the second factor, Greek characters or numbers levels of the third factor and Latin characters levels of the fourth factor. As has already been said, Graeco-Latin squares may be considered as designs with three types of blocks. Designs of Latin squares n × n require n^2 design points or observations, which is considerably less when compared to n^4 design points in full factorial experimental design.

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The following Graeco-Latin squares may be used for practical needs:

3×3

A1	B3	C2
B2	C1	A3
C3	A2	B1

4×4

A1	B3	C4	D2
B2	A4	D3	C1
C3	D1	A2	B4
D4	C2	B1	A3

5×5

A1	B3	C5	D2	E4
B2	C4	D1	E3	A5
C3	D5	E2	A4	B1
D4	E1	A3	B5	C2
E5	A2	B4	C1	D3

7×7

A1	B5	C2	D6	E3	F7	G4
B2	C6	D3	E7	F4	G1	A5
C3	D7	E4	F1	G5	A2	B6
D4	E1	F5	G2	A6	B3	C7
E5	F2	G6	A3	B7	C4	D1
F6	G3	A7	B4	C1	D5	E2
G7	A4	B1	C5	D2	E6	F3

8×8

A1	B5	C2	D3	E7	F4	G8	H6
B2	A8	G1	F7	H3	D6	C5	E4
C3	G4	A7	E1	D2	H5	B6	F8
D4	F3	E6	A5	C8	B1	H7	G2
E5	H1	D8	C4	A6	G3	F2	B7
F6	D7	H4	B8	G5	A2	E3	C1
G7	C6	B3	H2	F1	E8	A4	D5
H8	E2	F5	G6	B4	C7	D1	A3

9×9

A1	B3	C2	D7	E9	F8	G4	H6	I5
B2	C1	A3	E8	F7	D9	H5	I4	G6
C3	A2	B1	F9	D8	E7	I6	G5	H4
D4	E6	F5	G1	H3	I2	A7	B9	C8
E5	F4	D6	H2	I1	G3	B8	C7	A9
F6	D5	E4	I3	G2	H1	C9	A8	B7
G7	H9	I8	A4	B6	C5	D1	E3	F2
H8	I7	G9	B5	C4	A6	E2	F1	D3
I9	G8	H7	C6	A5	B4	F3	D2	E1

11×11

A1	B7	C2	D8	E3	F9	G4	H10	I5	J11	K6
B2	C8	D3	E9	F4	G10	H5	I11	J6	K1	A7
C3	D9	E4	F10	G5	H11	I6	J1	K7	A2	B8
D4	E10	F5	G11	H6	I1	J7	K2	A8	B3	C9
E5	F11	G6	H1	I7	J2	K8	A3	B9	C4	D10
F6	G1	H7	I2	J8	K3	A9	B4	C10	D5	E11
G7	H2	I8	J3	K9	A4	B10	C5	D11	E6	F1
H8	I3	J9	K4	A10	B5	C11	D6	E1	F7	G2
I9	J4	K10	A5	B11	C6	D1	E7	F2	G8	H3
J10	K5	A11	B6	C1	D7	E2	F8	G3	H9	I4
K11	A6	B1	C7	D2	E8	F3	G9	H4	I10	J5

12×12

A1	B12	C6	D7	I5	J4	K10	L11	E9	F8	G2	H3
B2	A11	D5	C8	J6	I3	L9	K12	F10	E7	H1	G4
C3	D10	A8	B5	K7	L2	I12	J9	G11	H6	E4	F1
D4	C9	B7	A6	L8	K1	J11	I10	H12	G5	F3	E2
E5	F4	G10	H11	A9	B8	C2	D3	I1	J12	K6	L7
F6	E3	H9	G12	B10	A7	D1	C4	J2	I11	L5	K8
G7	H2	E12	F9	C11	D6	A4	B1	K3	L10	I8	J5
H8	G1	F11	E10	D12	C5	B3	A2	L4	K9	J7	I6
I9	J8	K2	L3	E1	F12	G6	H7	A5	B4	C10	D11
J10	I7	L1	K4	F2	E11	H5	G8	B6	A3	D9	C12
K11	L6	I4	J1	G3	H10	E8	F5	C7	D2	A12	B9
L12	K5	J3	I2	H4	G9	F7	E6	D8	C1	B11	A10

Table 2.67 Analysis of variance of n × n Graeco-Latin square

Source of variations	f	SS	MS
Factor I rows	n-1	$SS_R = \frac{\sum_i Y^2_{i\bullet(\bullet\bullet)}}{n} - \frac{Y^2_{\bullet\bullet(\bullet\bullet)}}{n^2}$	$MS_R = \frac{SS_R}{n-1}$
Factor II columns	n-1	$SS_C = \frac{\sum_j Y^2_{\bullet j(\bullet\bullet)}}{n} - \frac{Y^2_{\bullet\bullet(\bullet\bullet)}}{n^2}$	$MS_C = \frac{SS_C}{n-1}$
Factor III latin characters	n-1	$SS_L = \frac{\sum_k Y^2_{\bullet\bullet(k\bullet)}}{n} - \frac{Y^2_{\bullet\bullet(\bullet\bullet)}}{n^2}$	$MS_L = \frac{SS_L}{n-1}$
Factor IV greek characters	n-1	$SS_G = \frac{\sum_l Y^2_{\bullet\bullet(\bullet l)}}{n} - \frac{Y^2_{\bullet\bullet(\bullet\bullet)}}{n^2}$	$MS_G = \frac{SS_G}{n-1}$
Experimental error	(n-1)(n-3)	$SS_E = \sum_i \sum_j \sum_k \sum_l Y^2_{ij(kl)} - \frac{Y^2_{\bullet\bullet(\bullet\bullet)}}{n^2} - SS_R - SS_C - SS_L - SS_G$	$MS_E = \frac{SS_e}{(n-1)(n-3)}$
Total	n^2-1	$SS_T = \sum_i \sum_j \sum_k \sum_l Y^2_{ij(kl)} - \frac{Y^2_{\bullet\bullet(\bullet\bullet)}}{n^2}$	–

The mathematical model of Graeco-Latin squares has the form:

$$y_{ij(kl)}=\mu+\alpha_i+\beta_j+\tau_k+\gamma_l+\varepsilon_{ij(kl)} \tag{2.53}$$

i, j, k, l=1, 2,..., n

where:

$$\sum \alpha_i = \sum \beta_j = \sum \tau_k = \sum \gamma_l = 0$$

$\varepsilon_{ij(kl)}=N\ (0, \sigma^2)$

Analysis of variance for Graeco-Latin squares is shown in Table 2.67.

Example 2.22 [21]

The subject of research is a change in technology of tempering copper pipes. The basic requirement to be observed when doing these changes is that minimal tensile strength should be 17 T/in^2. It has been suggested in the new technological procedure to temper copper pipes at a lower temperature and not to draw them out after tempering. The experiment has been designed exactly to research the suggested modification of the technological procedure. In defining the design of experiment one should take care of inequalities such as variations in material and the inside temperature of the furnace for tempering. Variations in quality of the initial material are accounted for by drawing out randomly eight copper pipes on each of eight days. These eight days are distributed within a three-week period with the idea of covering normal variations in quality of the observed technological procedure. In order to

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eliminate unequal distribution of furnace temperatures, the furnace has been divided into eight rows and eight columns, or into 64 shelves. Next, the experiment has been designed by Graeco-Latin squares with two factors:

- day of production;
- number added to each copper pipe in a sample of eight.

The factor number has no physical meaning and it has been included to enable identification of tensile strength results for each pipe at different tempering temperatures. In a different technological procedure these numbers might indicate the order of daily production or different procedures of preparing copper pipes, etc. Results of the experiment by a Graeco-Latin square are shown in Table 2.68. The table gives rows and columns as positions in the furnace, characters A to H as day of production, and numbers 1 to 8 as copper pipes in the sample. The results of measurements give tensile strength in tons per square inch.

Table 2.68 Design of experiment of 8 × 8 Graeco-Latin square

	1	2	3	4	5	6	7	8	Sum
1	16.6 D3	16.9 H4	17.4 C5	17.4 B6	15.8 E8	18.2 A1	15.7 G2	15.8 F7	133.8
2	15.9 F6	16.4 E5	15.8 G4	19.0 A3	17.6 H2	17.8 B7	18.9 C8	17.1 D1	138.5
3	17.1 B5	16.8 C6	19.2 H3	16.6 D4	15.8 G1	17.8 F8	18.4 E7	18.3 A2	140.0
4	17.7 A4	15.9 G3	16.3 E6	16.0 F5	17.6 C7	17.8 D2	18.1 H1	16.5 B8	135.9
5	17.4 C1	17.0 B2	16.8 D8	19.2 H7	20.3 A5	18.4 E3	15.9 F4	15.7 G6	140.7
6	16.5 E2	16.0 F1	16.9 A7	15.9 G8	17.1 D6	17.5 C4	17.4 B3	19.6 H5	136.9
7	15.8 G7	16.9 A8	15.9 F2	16.5 E1	17.6 B4	19.4 H6	17.1 D5	18.5 C3	137.5
8	18.6 H8	17.4 D7	17.4 B1	19.2 C2	16.8 F3	15.7 G5	17.4 A6	18.4 E4	140.9
Sum	135.6	133.3	135.7	139.8	138.6	142.6	138.9	139.7	1104.2

Table 2.69 Analysis of variance of 8 × 8 Graeco-Latin square

Source of variations	f	SS	MS	F	$F_{f;35;0.95}$
Between rows	7	543.2	77.6	1.47	2.29
Between columns	7	769.9	110.0	2.08	2.29
Between positions **	14	1313.1	93.8	1.77	1.98
Between days	7	4831.7	690.3	13.05	2.29
Between days *	7	322.2	46.0	0.87	2.29
Experimental error *	35	1852.9	52.9	–	–
Total	63	8319.9	–	–	–

As expected, the effect of differences between numbers is statistically unimportant and is of the same size as the experimental error. Hence we may join sums of squares for the factor between numbers and experimental error into a new combined experimental error*, with 42 degrees of freedom. Variations between columns

and rows in the furnace may also be combined, so that the effect between positions** in the furnace is at the limit of statistical significance. A high statistical significance has the effect of differences between copper pipes from day to day. This has been later on proved, since compositions of copper pipes in days F and G differed. As tensile strength level for tempering 300 °C with no later drawing out of all copper pipes has been below 17, the change in technological procedure cannot be accepted.

Example 2.23 [25]

The objective of this experiment is to define effects of the following factors on the corrosion rate of silicon rods: (1) decolorisation of nitric acid, (2) volume of corrosion matter, (3) size of silicon rod and (4) time spent in corrosion matter. The experiment went the like this: five bottles of recent nitric acid was exposed to different sunlight activity until a change in its color from colorless to light yellow occurred. The same kinds of nitric acid were used to produce a solution for corrosion. Five groups of silicon rods were sorted out by their weights. Five different volumes of corrosion matter and five corrosion times were used in the experiment. Order of doing the design point was random. The corrosion intensity measure is the loss in weight of silicon rods after rinsing and drying them. The results of the experiment are shown in Table 2.70.

Table 2.70 Design of experiment of 5 × 5 Graeco-Latin square

Volume of corrosion matter	Color of nitric acid				
	1	2	3	4	5
1	65 A1	82 B3	108 C5	101 D2	126 E4
2	84 B2	109 C4	73 D1	97 E3	83 A5
3	105 C3	129 D5	89 E2	89 A4	52 B1
4	119 D4	72 E1	76 A3	117 B5	84 C2
5	97 E5	59 A2	94 B4	78 C1	106 D3

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Results of analysis of variance are shown in Table 2.71.

Table 2.71 Analysis of variance of 5 × 5 Graeco-Latin square

Sources of variations	f	SS	MS	F	$F_{f;8;0.95}$
Color of acid	4	227.76	56.94	0.47	3.84
Volume	4	285.76	71.44	0.59	3.84
Size of rods	4	2867.76	716.94	5.96	3.84
Time of corrosion	4	5536.56	1384.14	11.50	3.84
Experimental error	8	962.72	120.34	–	–
Total	24	9880.56	–	–	–

■ **Problem 2.12 [25]**

Analyze the results of a 44 Graeco-Latin square.

Table 2.72 Design of experiment of 4 × 4 Graeco-Latin square

Rows	Columns				Sum
	1	2	3	4	
1	6 A1	4 B3	7 C4	5 D2	22
2	5 B2	6 A4	3 D3	4 C1	18
3	4 C3	5 D1	8 A2	4 B4	21
4	3 D4	2 C2	8 B1	6 A3	19
Sum	18	17	26	19	80

■ **Problem 2.13 [25]**

The data given below are results of 25 design points performed at five temperatures and with five different time periods, with the idea of establishing effects of the given factors on conversion in a chemical reactor. To avoid inequality effects, five chemical reactors and five operators were included in the experiment. So, 25 design points were done in five reactors with five operators by design of experiment of a 5×5 Graeco-Latin square in such a way that each operator used each reactor only once at each temperature and for a constant conversion time period. Characters denote reactors and numbers the operators. Do the analysis of variance.

Table 2.73 Design of experiment of 5 × 5 Graeco-Latin square

Temperature	Time					Sum
	30	60	90	120	150	
100	16 A1	40 B3	50 C5	20 D2	15 E4	141
125	30 B2	25 C4	62 D1	67 E3	30 A5	214
150	50 C3	50 D5	83 E2	85 A4	45 B1	313
175	80 D4	80 E1	95 A3	98 B5	70 C2	423
200	90 E5	92 A2	98 B4	100 C1	88 D4	468
Sum	266	287	388	370	248	1559

2.2.6 Youdens Squares

Observe the design of an incomplete random block with four blocks and four levels of researched factor.

This design is shown in Table 2.74.

Table 2.74 Design of experiment

Block	Factor		
1	A	B	C
2	D	C	A
3	B	A	D
4	C	D	B

This kind of design of experiment is known as *Youdens square* and is in essence an incomplete design of a Latin square as the fourth column with D factor levels in the first block, B in the second, C in the third and A in the fourth are missing. Design of experiment from Table 2.74 may be written differently, as shown in Table 2.75.

Table 2.75 Youdens design of experiment

Block	Factor			
	A	B	C	D
1	α	β	γ	–
2	γ	–	β	α
3	β	α	–	γ
4	–	γ	α	β

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The design of experiment written in this form is a reconstructed Latin square design where one of the diagonals has been left out. Generally speaking, *Youdens* square is a symmetrically balanced incomplete random block where each factor level appears once and only once in each block position.

Youdens square is always a Latin square where one or more columns (or rows or diagonals) have been left out; however, the opposite is not true; a Latin square where one or more columns (or rows or diagonals) have been left out is not always a Youdens square, for by leaving out columns from a Latin square the balance in design is lost. It is, however, possible to construct designs of Youdens squares from all symmetrical balanced random blocks [26]. Youdens squares have the same number of rows and levels of a researched factor but quite a different number of columns.

This notation is used:

1) I number of levels of researched factor;
2) J number of levels of one inequality source-blocks;
3) K number of levels of the second source of inequality;
4) L number of replications of each factor level

Following Youdens squares may be used for practical needs:

Table 2.76 I=J=7; K=L=3

Rows	Columns		
	1	2	3
1	G	A	C
2	A	B	D
3	B	C	E
4	C	D	F
5	D	E	G
6	E	F	A
7	F	G	B

Table 2.77 I=J=11; K=L=5

Rows	Columns				
	1	2	3	4	5
1	A	B	C	D	E
2	G	A	F	J	C
3	I	H	A	F	B
4	K	I	G	A	D
5	J	K	E	H	A
6	H	G	B	C	K
7	B	F	D	K	J
8	F	C	K	E	I
9	C	D	J	I	H
10	E	J	I	B	G
11	D	E	H	G	F

Table 2.78 I=J=7; K=L=4

Rows	Columns			
	1	2	3	4
1	D	F	G	A
2	E	G	A	B
3	F	A	B	C
4	G	B	C	D
5	A	C	D	E
6	B	D	E	F
7	C	E	F	G

Table 2.79 I=J=13; K=L=4

Rows	Columns												
	1	2	3	4	5	6	7	8	9	10	11	12	13
1	A	B	C	D	E	F	G	H	I	J	K	L	M
2	B	C	D	E	F	G	H	I	J	K	L	M	A
3	D	E	F	G	H	I	J	K	L	M	A	B	C
4	J	K	L	M	A	B	C	D	E	F	G	H	I

Table 2.80 I=J=15; K=L=7

Rows	Columns														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
2	B	C	D	E	F	G	H	I	J	K	L	M	N	O	A
3	C	D	E	F	G	H	I	J	K	L	M	N	O	A	B
4	E	F	G	H	I	J	K	L	M	N	O	A	B	C	D
5	F	G	H	I	J	K	L	M	N	O	A	B	C	D	E
6	I	J	K	L	M	N	O	A	B	C	D	E	F	G	H
7	K	L	M	N	O	A	B	C	D	E	F	G	H	I	J

Table 2.81 I=J=16; K=L=6

Rows	Columns															
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**
1	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
2	B	C	D	A	F	G	H	E	J	K	L	I	N	O	P	M
3	C	D	A	B	G	H	E	F	K	L	I	J	O	P	M	N
4	E	F	G	H	I	J	K	L	M	N	O	P	A	B	C	D
5	L	I	J	K	P	M	N	O	D	A	B	C	H	E	F	G
6	M	N	O	P	A	B	C	D	E	F	G	H	I	J	K	L

Table 2.82 I=J=16; K=L=10

Rows	Columns															
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**
1	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
2	C	A	B	E	F	D	J	I	H	G	M	K	L	P	N	O
3	D	C	A	K	M	G	H	E	L	I	J	B	P	O	F	N
4	N	E	P	A	H	B	D	C	F	K	O	G	I	J	L	M
5	M	N	O	P	B	A	F	D	E	C	G	I	J	H	K	L
6	B	J	H	G	A	I	L	O	M	N	D	C	E	F	P	K
7	L	K	I	B	O	P	N	A	D	F	C	H	G	E	M	J
8	J	H	F	L	G	M	A	P	K	O	B	N	C	D	E	I
9	I	P	L	O	N	K	C	M	J	A	H	E	F	B	D	G
10	O	M	K	J	L	N	P	G	A	E	F	D	B	I	C	H

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Youdens square I=J=7; K=L=4 is transformed into a balanced incomplete random block for easier calculation:

It should be noted that levels of second degree of inequality are given by numbers in brackets.

Table 2.83 Youdens 7 × 4 square

Factor	Blocks						
	1	**2**	**3**	**4**	**5**	**6**	**7**
A	(4)	(3)	(2)		(1)		
B		(4)	(3)	(2)		(1)	
C			(4)	(3)	(2)		(1)
D	(1)			(4)	(3)	(2)	
E		(1)			(4)	(3)	(2)
F	(2)		(1)			(4)	(3)
G	(3)	(2)		(1)			(4)

The previously introduced notation says:

- I number of levels of researched factor;
- J number of levels of one inequality source-blocks;
- K number of levels of the second source of inequality;
- L number of replications of each factor level.

Analysis of variance for Youdens squares is shown in Table 2.84.

Table 2.84 Analysis of variance for youdens square

Sources of variations	f	SS	MS	F
Blocks	I-1	$SS_1 = \frac{\sum_j Y^2_{\bullet j(\bullet)}}{K} - \frac{Y^2_{\bullet\bullet(\bullet)}}{JK}$	$\frac{SS_1}{I-1}$	$\frac{MS_1}{MS_E}$
Corrected factor	I-1	$SS_2 = \frac{I-1}{JK^2(K-1)} \sum_i \left(KY_{i\bullet\bullet} - \sum_{j(i)} Y_{\bullet j\bullet} \right)^2$	$\frac{SS_2}{I-1}$	$\frac{MS_2}{MS_E}$
Factor	J-1	$SS_3 = \frac{\sum_i Y^2_{i\bullet\bullet}}{L} - \frac{Y^2_{\bullet\bullet\bullet}}{IL}$	$\frac{SS_3}{J-1}$	$\frac{MS_3}{MS_E}$
Corrected blocks	J-1	$SS_4 = \frac{J-1}{JK^2(K-1)} \sum_j \left(LY_{\bullet j\bullet} - \sum_{i(j)} Y_{i\bullet\bullet} \right)^2$	$\frac{SS_4}{J-1}$	$\frac{MS_4}{MS_E}$
Second source of inequality	K-1	$SS_5 = \frac{\sum_l Y^2_{\bullet\bullet l}}{I} - \frac{Y^2_{\bullet\bullet\bullet}}{JK}$	$\frac{SS_5}{K-1}$	$\frac{MS_5}{MS_E}$
Experimental error	IK-J-I-K+2	$SS_E = \sum_i \sum_j \sum_k Y^2_{ijl} - \frac{Y^2_{\bullet\bullet\bullet}}{JK} - SS_1 - SS_2 - SS_5$	$\frac{SS_E}{IK-J-I-K+2}$	–
Total	IK-1	$SS_T = \sum_i \sum_j \sum_l Y^2_{ijl} - \frac{Y^2_{\bullet\bullet\bullet}}{JK}$	$\frac{SS_T}{IK-1}$	–

Note: $SS_1+SS_2=SS_3+SS_4$; I=J; K=L.

Example 2.24

In researching resistance on rubber abrasion, a *Martindale* tester is used for comparasions of various rubber samples. Five types of rubbers have been tested in five cycles on four tester positions. Design of experiment for five rubber types and five test cycles is the Youdens square shown in Table 2.85

Table 2.85 Youdens square

Cycle	Position on tester			
	α	β	γ	δ
1	A	B	C	D
2	E	A	B	C
3	D	E	A	B
4	C	D	E	A
5	B	C	D	E

Design of experiment and test results are shown in a more suitable form for calculations in Table 2.86.

Table 2.86 Youdens square

Rubber type treatment	Test cycle-block					Sum
	1	2	3	4	5	
A	68 α	33 β	54 γ	81 δ	–	236
B	49 β	31 γ	114 δ	–	40 α	234
C	80 γ	91 δ	–	56 α	50 β	277
D	71 δ	–	65 α	50 β	48 γ	234
E	–	51 α	49 β	70 γ	89 δ	259
Sum	268	206	282	257	227	1240

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I=5; J=5; K=4=L

$$SS_1 = \frac{268^2+206^2+282^2+257^2+227^2}{4} - \frac{1240^2}{5\times4} = \frac{311362.0}{4} - \frac{1537600}{20} = 960.50$$

$$SS_2 = \frac{5-1}{5\times4^2(4-1)}\left[(4\times236-268-206-282-257)^2 + \ldots + (4\times259-206-282-257-227)^2\right] = 719.50$$

$$SS_3 = \frac{\left(236^2+234^2+277^2+234^2+259^2\right)}{4} - \frac{1240^2}{5\times4} = 374.50$$

$$SS_4 = \frac{5-1}{5\times4^2(4-1)}\left[(4\times268-236-234-277-234)^2 + \ldots + (4\times227-234-277-234-259)^2\right] = 1305.5$$

$$SS_5 = \frac{(68+40+56+65+51)^2+\ldots+(81+114+91+71+89)^2}{5} - \frac{1240^2}{20} = 5273.20$$

$$SS_T = \left(68^2+33^2+54^2+81^2+\ldots+51^2+49^2+70^2+89^2\right) - \frac{1240^2}{20} = 85358-76880 = 8478$$

SS_E=8478-960.50-719.50-5273.20=1524.80

Complete the analysis of variance is shown in Table 2.87.

It is evident that factor and blocks have no statistically significant effect. However, between sample positions on the tester there exists a statistically significant difference. Position D gives much higher rubber abrasions, which may mean that samples of rubber have not been mounted properly in this position.

Table 2.87 Analysis of variance of Youdens square

Sources of variations	f	SS	MS	F
Between blocks	4	960.50	**reject**	–
Corrected factor	4	719.50	179.9	0.94
Factor	8	1680.00	–	–
Factor	4	374.50	**reject**	–
Corrected blocks	4	1305.50	326.4	1.71
Between blocks	8	1680.00	–	–
Between tester positions	3	5273.20	1757.7	9.22
Experimental error	8	1524.80	190.6	–
Total	19	8478.00	–	–

Example 2.25 [25]

Raw gasoline and six additives to be added into it were used to test the octane number of gasoline. Blocks are orders of testing, and columns are testing times. Design of experiment corresponded to a *Youdens* 73 square. Results of the experiment are shown in Table 2.88.

Table 2.88 Youdens 7 × 3 square

Blocks	Columns			Sum
	1	2	3	
1	43 A	34 B	47 D	124
2	36 B	32 C	46 E	114
3	33 C	47 D	43 F	123
4	44 D	40 E	33 G	117
5	41 E	35 F	44 A	120
6	36 F	32 G	32 B	100
7	33 G	41 A	27 C	101
Sum	266	261	272	799

Table 2.89 Youdens 7 × 3 square

Factor	Blocks							Sum
	1	2	3	4	5	6	7	
A	43(1)	–	–	–	44(3)	–	41(2)	128
B	34(2)	36(1)	–	–	–	32(3)	–	102
C	–	32(2)	33(1)	–	–	–	27(3)	92
D	47(3)	–	47(2)	44(1)	–	–	–	138
E	–	46(3)	–	40(2)	41(1)	–	–	127
F	–	–	43(3)	–	35(2)	36(1)	–	114
G	–	–	–	33(3)	–	32(2)	33(1)	98
Sum	124	114	123	117	120	100	101	799

For the sake of easier calculation Table 2.88 is transformed into Table 2.89.

$$SS_1 = \frac{124^2+114^2+123^2+117^2+120^2+100^2+101^2}{3} - \frac{799^2}{7\times 3} = 196.95$$

$$SS_2 = \frac{7-1}{7\times 3^2(3-1)}\left[(3\times 128-124-120-101)^2\right.$$

$$\left.+\ldots+(3\times 98-117-100-101)^2\right] = 493.62$$

$$SS_3 = \frac{128^2+102^2+92^2+138^2+127^2+114^2+98^2}{3} - \frac{799^2}{7\times 3} = 608.29$$

$$SS_4 = \frac{7-1}{7\times 3^2(3-1)}\left[(3\times 124-128-102-138)^2\right.$$

$$\left.+\ldots+(3\times 101-128-92-98)^2\right] = 82.29$$

$$SS_5 = \frac{(43+36+33+44+41+36+33)^2+\ldots+(44+32+27+47+46+43+33)^2}{7} - \frac{799^2}{7\times 3} =$$

$$= \frac{266^2+261^2+272^2}{7} - 30400.05 = 8.67$$

$$SS_T = \left(43^2+44^2+41^2+34^2+\ldots+36^2+33^2+32^2+33^2\right) - \frac{799^2}{7\times 3} = 706.95$$

SS_E=706.95-196.95-493.62-8.67=7.71

Complete analysis of variance is shown in Table 2.90.

Analysis of variance shows that addition of additives to basic gasoline has a statistically significant effect. The order of testing gasoline or differences between blocks are also statistically very important. The time factor is not statistically significant.

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Table 2.90 Analysis of variance of youdens square

Sources of variations	f	SS	MS	F	$F_{f;6;0.95}$
Order-blocks	6	196.95	–	–	–
Gasoline type-corrected factor	6	493.62	82.27	64.27	4.28
Gasoline type-factor	6	608.29	–	–	–
Order-corrected blocks	6	82.29	13.72	10.72	4.28
Time	2	8.67	4.34	3.39	5.14
Experimental error	6	7.71	1.28	–	–
Total	20	706.95	–	–	–

■ **Problem 2.14 [25]**

On the machine for abrasion testing four samples of dyestuff were tested. The machine has three positions for testing in the same time simultaneously. Tests have been made in four cycles. The design of experiment was *Youdens* 43. Results are shown in the next table. Do analysis of variance.

Table 2.91 Youdens square

Positions	Cycles			
	1	2	3	4
1	13 A	20 D	23 C	21 B
2	15 B	10 A	26 D	29 C
3	18 C	11 B	15 A	28 D

■ **Problem 2.15**

Seven procedures of a critical material state that their product satisfies very precise tensile strength requirements. Each producer has supplied enough material for four tests. Testing this material has been designed under seven conditions. Tensile strength tests have been done on four testers (1, 2, 3 and 4). Testing has been done by *Youdens* square design. Process the outcomes of this testing by analysis of variance.

Table 2.92 Design of experiment

Conditions	Producers							Sum
	1	2	3	4	5	6	7	
1	1.62(4)	2.10(3)	1.50(2)	–	2.30(1)	–	–	7.52
2	–	1.93(4)	1.90(3)	1.77(2)	–	1.64(1)	–	7.24
3	–	–	2.22(4)	1.56(3)	2.29(2)	–	1.92(1)	7.99
4	2.14(1)	–	–	1.58(4)	1.88(3)	1.81(2)	–	7.41
5	–	1.65(1)	–	–	1.61(4)	1.65(3)	2.03(2)	6.94
6	1.99(2)	–	1.64(1)	–	–	1.74(4)	2.46(3)	7.83
7	1.75(3)	2.46(2)	–	1.86(1)	–	–	2.62(4)	8.69
Sum	7.50	8.14	7.26	6.77	8.08	6.84	9.03	53.62

Summary

This chapter about design of experiments refers to screening the factors by the significance of their effect on a measured value or response. The researcher has by applying the mentioned methodology:

- defined all factors and responses of the research subject;
- defined all variable parameters of the research subject as random values;
- clarified the random property of the research subject variables and checked whether their distribution laws correspond to a normal distribution;
- estimated correlation between variables of the research subject;
- that factors, in accord with preliminary information, are ranked according in order of their effects on response by using a preliminary ranking method;
- screened out factors into significant and random ones as regards their affect on the research subject, by the random balance method.

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Based on the results of a screening experiment and the objective of the research problem, the researcher decides about including a system factor and response into the design of the basic experiment. This does not involve all the information of a selective experiment:

- Information on factor-variation intervals may be drawn from an active experiment by the random balance method. Thus, linear effects of factors X_1X_2 in Example 2.10 considerably exceed the affects of other factors. This simultaneously may mean that the selected factor-variation intervals $X_1(-;+)$; $X_2(-;+)$ are too high. If this is so, then they should be cut in half in the basic experiment.
- Factor space may be obtained from the matrix of random balance and analysis of variance. Information on number of replications of design points-trials in the basic experiment is obtained from analysis of variance, and some proofs about linear or nonlinear relationships between variables of the research subject from correlation analysis.

Hence, a more complete conclusion on all that is obtained from screening experiments should include:

- factor space;
- factor-variation intervals;
- preliminary number of design points/trials and replications;
- number of factors and their interactions, which should be included into the basic experiment.

2.3 Basic Experiment-Mathematical Modeling

General form of mathematical model for a research subject has been defined in Sect. 2.1.1:

$$y = \varphi(X_1, X_2, ..., X_k)$$

To select a mathematical model means to choose a form of mathematical function and write down the associated equation. The next step is designing and performing the experiment based on the results of which constants or coefficients of the chosen mathematical model will be determined. Choice of mathematical model is the next question. To obtain a satisfactory answer, let us observe the geometrical interpretation of this model or *response functions*. The mentioned response function geometrically is often called the *response surface*. In cases when the response is a function of several factors, the possibility of geometrical interpretation is lost. We are entering the area of abstract multidimensional space where it is difficult to get oriented. In such cases we switch to the language of algebra where computers help us find our way much more easily.

Let us stick to response geometrical interpretation of "black box" with two input factors. A simple graphic system with x-y coordinates is sufficient for this. One may insert values of variation levels of one factor on one axis, and those of the other factor on another axis. Each "black box" status will have a corresponding point in the surface. As has been said in Sect. 2.1.3, factors are defined by their domains. This means that each factor is defined by its minimal and maximal values where it may be changed continuously or discontinuously. If the factors are concordant then those limits in the plane form a rectangle within which are the points that coincide with "black box" statuses. Dashed lines in Fig. 2.28 mark the limit values of the *domain of factors* and full lines the limits of *concordant domain of factors*. To present graphically the response values, we use the third axis of the coordinate system, so that the response surface has the shape given in Fig. 2.29.

The area where the response surface has been constructed is called the *factor space*. The area taken by factor axes is often considered as the factor space. A response function does not have to be geometrically interpreted in a three-dimensional space for a research subject defined by only two factors. For such a presenta-

Hence, a more complete conclusion on all that is obtained from screening experiments should include:

- factor space;
- factor-variation intervals;
- preliminary number of design points/trials and replications;
- number of factors and their interactions, which should be included into the basic experiment.

2.3 Basic Experiment-Mathematical Modeling

General form of mathematical model for a research subject has been defined in Sect. 2.1.1:

$$y = \varphi(X_1, X_2, ..., X_k)$$

To select a mathematical model means to choose a form of mathematical function and write down the associated equation. The next step is designing and performing the experiment based on the results of which constants or coefficients of the chosen mathematical model will be determined. Choice of mathematical model is the next question. To obtain a satisfactory answer, let us observe the geometrical interpretation of this model or *response functions*. The mentioned response function geometrically is often called the *response surface*. In cases when the response is a function of several factors, the possibility of geometrical interpretation is lost. We are entering the area of abstract multidimensional space where it is difficult to get oriented. In such cases we switch to the language of algebra where computers help us find our way much more easily.

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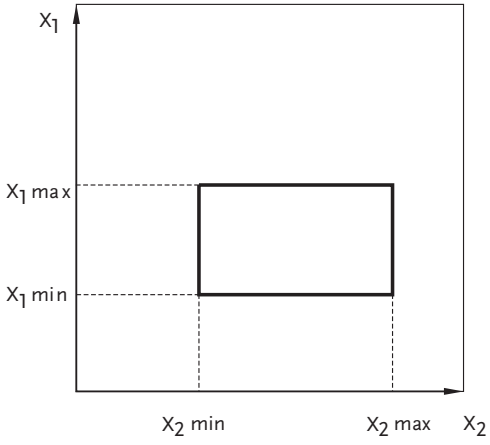

Figure 2.28 Factor space

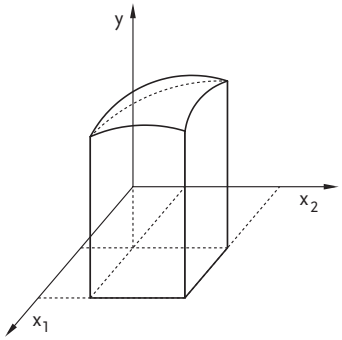

Figure 2.29 Response surface

tion a space where intersection lines of response surface are projected with planes parallel to X_10 X_2 is sufficient. This is shown in Fig. 2.30.

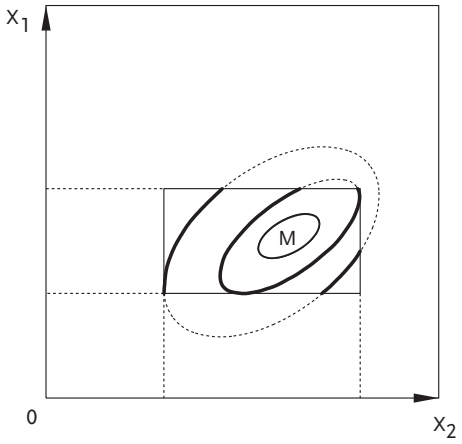

Figure 2.30 Contour lines

Point M in the figure is the optimum that by one definition of the research problem objective should be determined. Each intersection line in the plane is a line of constant response values and is called *contour lines-contour diagram*.

Since the way of interpreting a response function is clear, the basic question of finding the optimum with minimal expenses are as follows:

- When all possible response values are available for all combinations of factor variation levels in the form of a table, there is no problem to define or select

the model, nor is there a problem to find the optimum. An optimum is the combination of factors that has the best response value. As there are endless combinations of factors, this case is of no practical value.

- The second approach consists of choosing a random number of combinations of factor levels and their responses hoping that an optimal value has been included or in a limit case it is a value close to the optimal. Evidently, such an approach includes a great risk.
- The third approach involves mathematical modeling or obtaining a mathematical model by which response values outside the region of the experiment may be extrapolated. Response values that are an optimum or close to it are estimated.

Lack of all possible response values, for all possible combinations of factor-variation levels is paid by introduction of assumptions about an unknown model for use before the start of an experiment. The main assumptions about the model or response surface refer to continuity, smoothness and existence of an optimum. Such assumptions allow approximations of response functions by a polynomial in the vicinity of any factor-surface point. In the case of one optimum, the way we approach it is not essential, but if there are more, the problem becomes harder. Fig. 2.31 shows two response functions of one factor. The first one exactly corresponds to the introduced assumptions, and the second one is a case where assumptions about smoothness and continuity are broken, for the optimum and peak exist. If in searching for an optimum in such a function we start gradually from one side, we shall find the smaller maximum not knowing that a bigger one exists too. If we know response values in several neighboring points of a factor space, it is possible to estimate values of the same response in other neighboring points. We may successively find such points where the biggest increase (or decrease if a minimum is looked for) of a response value is expected. Then it becomes clear that the next experiment should be moved into those very points. This means that we should move into that direction neglecting others. By introducing a new experiment, we may determine the direction of fastest response changes again after processing the outcomes.

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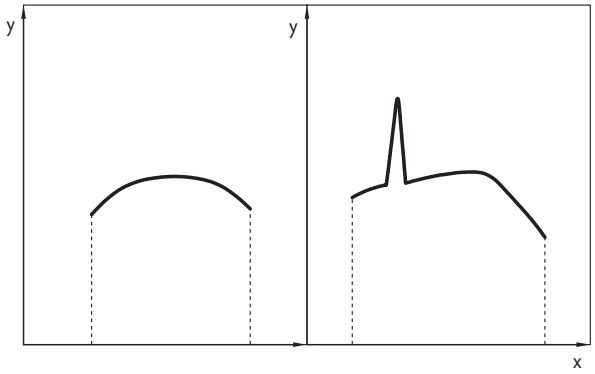

Figure 2.31 Response functions

To put it more clearly, we select a point and a cluster of points around it in the factor space, that is, we choose a subfactor space in it. The experiment is done in that region and based on it the first model is defined. Such a model is used to estimate the response outside the experimental region. Such an estimation of a response is called *extrapolation*. If the same is done for points within the experimental region it is then called *interpolation*. Since extrapolation confidence diminishes with distance from this region, it is done in its vicinity. Extrapolation outcomes are used to choose conditions for performing the next experiment. A further procedure in finding the optimum is repeated. To choose a model for doing the first so-called *basic experiment*, it is necessary for the model to fulfill certain requirements.

The analyzed approach to finding the optimum indicates that the model must have a possibility to estimate the direction of further design points with associated precision. Since we are unaware of the direction of movement towards the optimum before defining the model, the accuracy of estimation of the mathematical model must be the same in all directions. This is to say that estimated values in experiment region may differ from the real, measured response values for the previously given magnitude. The model which fulfills this requirement is called *adequate*, to be discussed in a separate section.

When several mathematical models fulfill the requirements, then, in principle, a simpler model is accepted. For us, it is the polynomial model that in the case of two factors may have these forms:

- Polynomial of the null order:

$$y=b_0 \tag{2.54}$$

- Polynomial of the first order:

$$y = b_0 + b_1 X_1 + b_2 X_2 \tag{2.55}$$

- Polynomial of the second order:

$$y = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2 + b_{11} X_1^2 + b_{22} X_2^2 \tag{2.56}$$

- Polynomial of the third order:

$$y = b_0 + b_1X_1 + b_2X_2 + b_{12}X_1X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{112}X_1^2X_2 + b_{122}X_1X_2^2 + b_{111}X_1^3 + b_{222}X_2^3 \quad (2.57)$$

This means that we have presented the unknown response function by a polynomial of the corresponding order. The operation of replacing one function with another one is called *approximation*. Since a polynomial may be of a different order, it is necessary to select a polynomial order for the basic experiment or for the first step towards the optimum. The experiment should determine numerical values of the polynomial coefficient. The valid rule here is: the higher the polynomial order the more coefficients or design points in the experiment exist. As the objective of a designed experiment is a minimum of design points, a polynomial of the first-order is selected for the basic experiment. The first order polynomial contains the information on the *gradient* direction or the direction of the fastest response change, and it has, at the same time, the smallest number of coefficients. The only questionable thing is whether the linear model will always be adequate. It is however, known in mathematics that each point has an environment where that model is adequate. The only remaining thing is selection of the factor subdomain where the linear model is adequate. The size of that subdomain is not known, but lack of fit of the model may be checked by outcomes of the experiment. Hence, an arbitrary subdomain is selected in advance, to be corrected later to the corresponding size after checking its lack of fit.

In this way the basic experiment is defined for the linear model, and the gradient that indicates the direction of the fastest response increase or decrease is obtained. When a response maximum or minimum is searched for, the experimental center is moved that way and a new experiment for the linear model performed. The procedure is repeated until moving along the gradient has an effect. When this has no effect, it means we are close to the optimum. Polynomials of higher order, mostly the second, are used in the optimum region.

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All this clearly shows that selection of the subdomain for performing the basic experiment is very important and it will be discussed in the coming sections.

Apart from optimization, a problem is often set for mathematical modeling or interpolation. The optimum does not interest us in that case but the model that adequately describes the obtained results in the experimental field. A subdomain is not chosen in that case, but the polynomial order is moved up until an adequate model is obtained. When a linear or incomplete square model (with no members with a square factors) is adequate it means that the research objective corresponds to the optimization objective.

Summary

Previous considerations have given a model that will be systematically analyzed when designing a basic experiment. It is the algebraic polynomial of the first-order or linear model.

To select it, it has been necessary to analyze-geometric interpretation of the response surface in the factorial domain. The response surface is defined in the

associated factor space only. In this domain, one response value corresponds to any combination of factors or state of the research subject. A response surface, apart from its interpretation in a Cortesian coordinate system, may also be presented in a plane in the form of a response contour diagram. It has been mentioned that the mathematical model in the first phase of experimenting serves to determine the gradient or direction of the fastest response change. A model in the form of an analytical function with one optimum has been adopted. Since the analytical function is in question, it may be approximated by a polynomial around any point in the factor space.

Accepting the given assumptions, finding an optimum, as the most complex problem, is reduced to the following iterative procedure: set up the experiment with a minimum of design points and from its outcomes determine the model base on which the gradient is obtained, and the procedure is repeated in that direction until an optimum is reached. The model must meet the requirements defined by its lack of fit and simplicity. Adequacy or lack of fit of the model means estimating experimental response values with the necessary accuracy in the experimental region. Lack of fit of the model is checked after performing the experiment. As a simple model, a polynomial of the first order has been selected, which is linear by its unknown coefficients and determined by processing experimental results. The linear polynomial has been selected as it offers a small enough experimental region where such a model is adequate. Choice of experimental region depends on the researcher and it is not completely formalized. In the case of having to solve the problem of modeling or interpolation and not optimization, the experimental region is fixed beforehand and the procedure completed by obtaining an adequate model in that region. When a linear model is inadequate, the polynomial level is increased until the condition of lack of fit is fulfilled.

2.3.1
Full Factorial Experiments and Fractional Factorial Experiments

When designing the basic experiment, the unknown response function (2.5) is, in principle, approximated by a polynomial of the corresponding degree (2.6) where regression coefficients are estimated on the basis of experimental results (2.7). A linear mathematical model is considered in the first phase of a research. Defining the first order regression model is the first phase of a study objectiveed at obtaining the interpolation model or function, the knowledge of which facilitates estimating response values in different points of the studied factorial space. A linear model is, additionally, also used when moving to the optimum region, the same as when we use the steepest ascent method as an optimization technique. Later, if necessary, the polynomial degree is increased. Lack of fit of polynomial models is checked by methods of statistical analysis.

In defining a linear model, coefficient b_0 and all linear regression coefficients are calculated.

$$\widehat{y} = b_0 + \sum_{i}^{k} b_i X_i + \sum_{i}^{k} b_{ij} X_i X_j \tag{2.58}$$

where:

$\widehat{y}$ is response value;

b_i are linear regression coefficients;
b_{ij} are regression coefficients of double factor interactions.

Accuracy and confidence of the obtained estimates for regression coefficients depend on the used design of experiments. Choice of the design of experiments has to do with determining the number of experimental points-trials and such a distribution of those points in a factorial space that facilitates obtaining the necessary information with a minimal number of design points-trials. When selecting the *design of experiments* a *design matrix* or a standard type table is constructed where all conditions of doing design points that are part of the chosen design are defined. Mostly, in a design matrix, rows correspond to different design points-trials and columns to individual factors. Obtaining a linear model has to do with performing *full factorial experiment-FUFE* or *fractional factorial experiment-FRFE*, which is a definite part of *FUFE*. *FUFE* is the experiment where all possible combinations of levels of factors are realized and experimental results are processed by applying statistical analysis. The number of *FUFE* design points-trials is determined from relation (2.20) where one should know that factors are varied on two levels in *FUFE*. *FUFE* is therefore called the design experiment of the type 2^k. In the case of the large number of factors (k), *FUFE* requires a large number of trials ($N=2^k$), so that in that case *FRFE* is used more frequently. The *FRFE* design matrix is called a *fractional replica*. When composing *FUFE* and *FRFE* matrices *coded* factor values are used. Coding factors requires linear transformation of the factor space coordinates with the coordinate beginning in the *null point* or *experimental center* and defining the coordinate axes ratio in units of the factor variation interval. The arithmetic of this transformation is given in this expression:

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$$X_i = \frac{x_i - x_{i0}}{\Delta x} \tag{2.59}$$

where:
X_i is the coded value of the i-th factor (nondimensional magnitude);
x_i, x_{i0} are natural or the real factor values, their current and null values, respectively;
$\Delta x(\varepsilon)$ is the natural value of the factor-variation interval. In a design matrix when we vary factors on two levels (+1; -1) only signs (+;-) exist.

With *FRFE* we differentiale the existence of *regular* and *irregular replicas*. A regular fractional replica is obtained from a *FUFE* matrix by dividing it into a number of parts divisible by two (2, 4, 6, 8, 16, etc.). The obtained replicas are respectively marked by: 1/2-replica or half-replica, 1/4-replica, 1/8-replica, etc. Irregular replicas are obtained by taking, for example 3/4 and 5/8 of the *FUFE* matrix.

Let us now consider how to construct *FUFE* and *FRFE* matrices. The number of trials is defined in the first column of the *FUFE* design matrix. The next column is a fictional variable ($x_0 = +1$) that is used to estimate the b_0 free member in the regression equation. The number of matrix columns that corresponds to the number of factors. Sometimes columns which correspond to factor interactions are also

obtained. The number of rows equals the number of design points-trials and is determined as $N=2^k$. An example of *FUFE* design matrix construction is shown in Table 2.93. Note that the column of the *fictional factor* is often dropped. For any number of factors k, the *FUFE* design matrix is constructed so that the matrix for (k-1) factors is repeated twice; first for the lower-level k factor values, and then for the upper-level ones. Distribution of experimental or design points-trials in factor space for *FUFE* and k=2; k=3 is shown in Figs. 2.32 and 2.33.

Table 2.93 FUFE design

No. trials	X_0	X_1	X_2	X_3	X_4	X_5
1	+	−	−	−	−	−
2	+	+	−	−	−	−
3	+	−	+	−	−	−
4	+	+	+	−	−	−
5	+	−	−	+	−	−
6	+	+	−	+	−	−
7	+	−	+	+	−	−
8	+	+	+	+	−	−
9	+	−	−	−	+	−
10	+	+	−	−	+	−
11	+	−	+	−	+	−
12	+	+	+	−	+	−
13	+	−	−	+	+	−
14	+	+	−	+	+	−
15	+	−	+	+	+	−
16	+	+	+	+	+	−
17	+	−	−	−	−	+
18	+	+	−	−	−	+
19	+	−	+	−	−	+
20	+	+	+	−	−	+
21	+	−	−	+	−	+
22	+	+	−	+	−	+
23	+	−	+	+	−	+
24	+	+	+	+	−	+
25	+	−	−	−	+	+
26	+	+	−	−	+	+
27	+	−	+	−	+	+
28	+	+	+	−	+	+
29	+	−	−	+	+	+
30	+	+	−	+	+	+
31	+	−	+	+	+	+
32	+	+	+	+	+	+

As the figures show, points of the design 2^2 are given by the square apex coordinates, and design points of the design 2^3 by the cube apex coordinates. Design points for k>3 are distributed in an analogous way. Regression coefficients of Eq. (2.58) are determined from *FUFE* outcomes. For k=3, these coefficients are determined:

$$\hat{y} = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{123}X_1X_2X_3 \tag{2.60}$$

b_{123}	regression coefficient of triple interaction;
b_{12},b_{13} ,b_{23}	regression coefficients of double interactions;
b_1,b_2,b_3	linear regression coefficients;
b_0	free member.

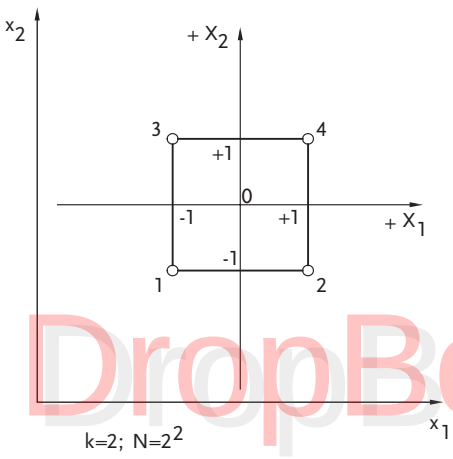

Figure 2.32 Distribution of points in factor space

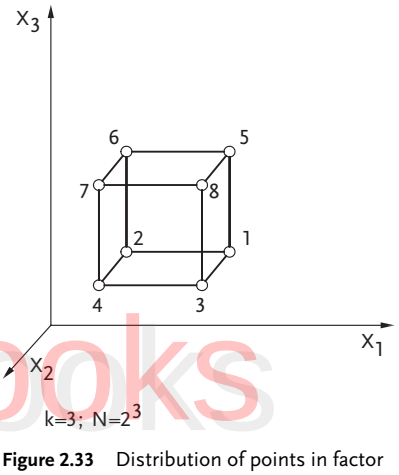

Figure 2.33 Distribution of points in factor space

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It has already been said that based on the magnitude of the linear regression coefficients one may speak about the strength of influence of associated factors on response. The higher the b_i value of the associated factor, the more intensively it affects response. The sign of those coefficients has to be accounted for too; if b_i has a positive sign, the increase of the associated factor causes an increase in response; on the contrary, with a negative sign of the linear regression coefficient an increase in its factor value causes a decrease in the optimization parameter. It is sometimes more interesting to observe the effect of the i-th factor (its value is equal to $2b_i$) and that way estimate the effect of the i-th factor on response as it changes from lower (-1) to upper (+1) level. This is especially applied in designs that include qualitative factors. The total number of all possible effects that may be calculated from *FUFE* corresponds to the number of N design points-trials. The number of linear regression coefficients is identical to the number of k factors, included in the *FUFE* matrix. To determine the number of factor interactions we use the formula:

$$C_k^m = \frac{k(k-1)(k-2)\ldots(k-m+1)}{1\times 2\times 3\times \ldots \times m} \tag{2.61}$$

where:
k is number of factors;
m is number of factors in interaction or an interaction order.

When analyzing results of factorial experiments we talk about *main effects* and *interaction effects*. Main effects are factor effects and they are the difference of averaged response for two levels (+1; -1) for the associated factor. In case response difference for two levels of factor X_1 is the same irrespetive of on which level factor X_2 (excluding experimental error), one may say that there exists no interaction between factors X_1 and X_2 or that the interaction is X_1X_2=0. This statement may be graphically presented. Figures 2.34 and 2.35 show interaction between factors X_1 and X_2, and Fig. 2.36 indicates that such an interaction is nonexistent.

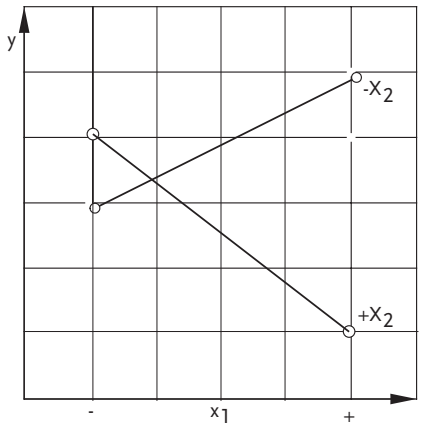

Figure 2.34 Response values indicate existence of interaction X_1X_2

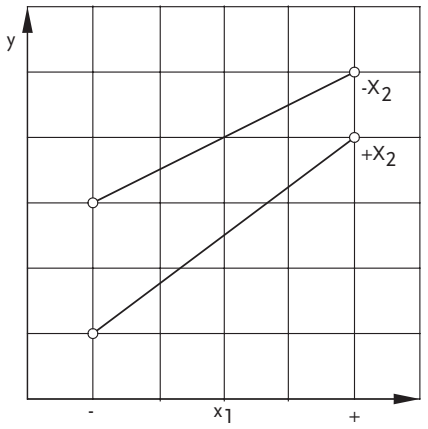

Figure 2.35 Response values indicate existence of interaction X_1X_2

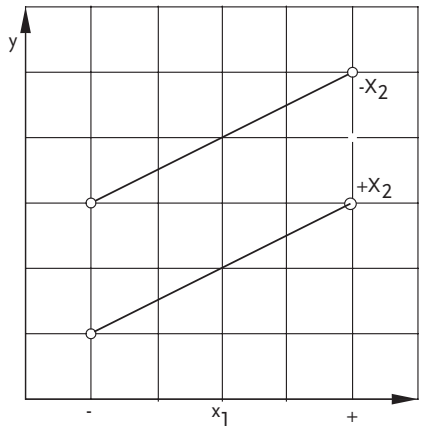

Figure 2.36 Response values indicate nonexistence of interaction X_1X_2

It is frequently the case in practice that certain interactions are statistically unimportant and those are mostly interactions of a higher order (triple, quadruple, etc.). This property of higher-order interactions has precisely been used for constructing fractional replicas. A fractional design is obtained so that statistically unimportant interactions are replaced in a *FUFE* column design by a new factor. Here, it should be pointed out that when forming a fractional replica, *FUFE* rows are not mechanically divided. A *FUFE* design may not be simply divided into two parts when we form a half-replica. Fractional replicas or designs must be formed in accord with the rule of *design saturation*. A saturated design denotes the replica that is obtained when replacing all interaction effects with linear effects of new factors so that the degree of freedom f=0. The experiment in this case involves a minimal number of design points, and the outcomes may result in erroneous conclusions if the linear model is inadequate and if interaction effects significantly affect the linear-effect estimates. A check of lack of fit of linear models, obtained from saturated designs, is not feasible as the degree of freedom is f=0.

Taking all this into consideration, unsaturated designs (f>0) or special designs, which include the influence of interaction effects on linear-effect estimates, are used in practice. An oversaturated design (f<0) was used in Example 2.12 as a random balance method design, but a totally different problem was being solved in that case.

To compare estimates of efficiency of fractional replicas, a special criterion called *power of solving a replica* is used. This criterion includes a number of linear effects that have not been aliased/confounded in the given design. In the case of aliased/confounded effects, we obtain aliased/confounded regression coefficients when processing experimental outcomes, which simultaneously characterize both linear and interaction effects. By mixing the effects on this way, the number of design points-trials is reduced but at the same time the analysis of experimental outcomes is complicated. From the same *FUFE* design one may get a replicas with different degrees of effect mixing or a different power of solving them. A researcher, however, should, in principle, try to find the replica with the highest possible power of solving it. This is most often achieved when new linear effects are aliased/confounded with highest-order interactions. This is exactly the principle used in fractional replicas in Example 2.12 to form the oversaturated design of the random balance method. Namely, a replacement of triple interactions by a new factor $X_1X_2X_3=X_4$ has been done in that design. Higher-order interactions are replaced by new linear effects because of the fact that we may assume, with a high level of confidence, that those interactions are less significant than lower-order interactions. Simultaneously, a probability of obtaining nonaliased/confounded linear effects increases. An estimate of the power of solving a replica is additionally a complicated case of several factorial experiments. To alleviate the problem two new terms are introduced: *generating ratio* and *defining contrast*.

Generating ratio is the term that indicates the effect with which the new effect is aliased/confounded. Actually, when replacing a triple interaction $X_1X_2X_3$ by X_4 factor, the generating ratio has the form $X_1X_2X_3=X_4$. By increasing the number of symbols in a generating ratio the power of solving a replica is increased. There is a higher power of solving a replica in the example $X_1X_2X_3=X_4$ than in the case of $X_1X_2=X_4$.

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Defining contrast is obtained by multiplying the generating ratio by its associated factor. The defining contrast, the case, $X_1X_2X_3=X_4$ has the form: $1=X_1X_2X_3X_4$.

The given ratio helps to determine aliased/confounded effects. For this, it is necessary to multiply successively both sides of the defining contrast by factors from matrix columns. Factor X_4 is in this case obtained:

$$X_4=X_1X_2X_3X_4^2=X_1X_2X_3$$

This system of mixing effects may conveniently be written as regression coefficients. For factor X_4 in this case we obtain:

$$b_4=\beta_4+\beta_{123}$$

For other factors and interactions we get:

$$b_1=\beta_1+\beta_{234} \quad ; \quad b_2=\beta_2+\beta_{134} \quad ; \quad b_3=\beta_3+\beta_{124};$$
$$b_{12}=\beta_{12}+\beta_{34}; \quad b_{13}=\beta_{13}+\beta_{24}; \quad b_{23}=\beta_{23}+\beta_{14}$$

Fractional replicas may also be observed as designs of the type $2^{k\text{-}p}$, where p is the number of linear effects aliased/confounded with interaction effects. As has been said for construction of random balance design, fractional replica $2^{4\text{-}1}$ has been used in Example 2.12. Design of type $2^{3\text{-}1}$ means two replicas as defined by two generating ratios $X_1X_2=X_3$ and $-X_1X_2=X_3$. For the first half-replica, factor X_3 replaces in the design matrix the column that corresponds to X_1X_2, Table 2.94.

Table 2.94 Fractional factorial design 2^{3-1}

No. trials	X_0	X_1	X_2	$X_3=X_1X_2$
1	+	+	+	+
2	+	−	+	−
3	+	+	−	−
4	+	−	−	+

Signs in the interaction column are obtained by simply multiplying the columns of associated factors.

By multiplying the chosen generating ratio with new factor X_3 we obtain the defining contrast: $1=X_1X_2X_3$.

It is then multiplied by each factor from the design $2^{3\text{-}1}$. If the given yields offer the square of the factor, it is automatically replaced by the number one. Aliased/confounded effects for the observed half-replica are given by these ratios:

$$X_1=X_2X_3; \quad X_2=X_1X_3; \quad X_3=X_1X_2$$

This means that regression coefficients will be estimated as these aliased/confounded effects:

$$b_1=\beta_1+\beta_{23}; \quad b_2=\beta_2+\beta_{13}; \quad b_3=\beta_3+\beta_{12};$$

To illustrate this let us observe the *FUFE* 2^5. In this case we obtain a half-replica of type 2^{5-1}, as given by the generating ratio $X_5=X_1X_2X_3X_4$. The associated defining contrast is: $1=X_1X_2X_3X_4X_5$, and aliased/confounded estimates are defined by these ratios:

$$\begin{array}{lll} X_1 = X_2X_3X_4X_5; & X_1X_2 = X_3X_4X_5; & X_2X_4 = X_1X_3X_5; \\ X_2 = X_1X_3X_4X_5; & X_1X_3 = X_2X_4X_5; & X_2X_5 = X_1X_3X_4; \\ X_3 = X_1X_2X_4X_5; & X_1X_4 = X_2X_3X_5; & X_3X_4 = X_1X_2X_5; \\ X_4 = X_1X_2X_3X_5; & X_1X_5 = X_2X_3X_4; & X_3X_5 = X_1X_2X_4; \\ X_5 = X_1X_2X_3X_4; & X_2X_3 = X_1X_4X_5; & X_4X_5 = X_1X_2X_3; \end{array}$$

Now consider the *FUFE* 1/16-replica for eight factors. In this case the design of type 2^{8-4} is defined by four generating ratios:

$X_5=X_1X_2X_3X_4$; $X_6=X_1X_2X_3$;
$X_7=X_1X_2X_4$; $X_8=X_1X_3X_4$;

The design matrix is shown in Table 2.95.

Table 2.95 Fractional factorial design 2^{8-4}

No. trials	Design matrix									Response
	X_0	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	$\bar{y}_u$
1	+	+	+	+	+	+	+	+	+	18.1
2	+	+	+	−	+	−	−	−	+	32.1
3	+	+	−	+	+	−	−	+	−	19.5
4	+	+	−	−	+	+	+	−	−	20.5
5	+	−	+	+	+	−	−	+	+	15.0
6	+	−	+	−	+	+	+	−	+	25.8
7	+	−	−	+	+	+	+	+	−	24.0
8	+	−	−	−	+	−	−	−	−	43.8
9	+	+	+	+	−	−	+	−	−	25.0
10	+	−	−	−	−	+	−	+	+	52.0
11	+	−	+	+	−	+	−	−	−	42.6
12	+	+	−	−	−	−	+	+	+	29.2
13	+	+	−	+	−	+	−	−	+	25.0
14	+	−	+	−	−	−	+	+	−	55.0
15	+	+	+	−	−	+	−	+	−	45.0
16	+	−	−	+	−	−	+	−	+	34.0

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Defining contrasts are:

$1=X_1X_2X_3X_4X_5$; $1=X_1X_2X_3X_6$;
$1=X_1X_2X_4X_7$; $1=X_1X_3X_4X_8$;

By multiplying defining contrast (two, three or four) we obtain the general defining contrast:

$$1=X_1X_2X_3X_4X_5=X_1X_2X_3X_6=X_1X_2X_4X_7=X_1X_3X_4X_8=X_4X_5X_6=X_3X_5X_7=X_2X_5X_8=$$
$$=X_3X_4X_6X_7=X_2X_4X_6X_8=X_2X_3X_7X_8=X_1X_2X_5X_6X_7=X_1X_3X_5X_6X_8=X_1X_6X_7X_8=$$
$$=X_1X_4X_5X_7X_8=X_2X_3X_4X_5X_6X_7X_8.$$

By neglecting all higher-order interactions starting with triple ones, we obtain these estimates of regression coefficients:

$b_1=\beta 1$; $b_2=\beta_2+\beta_{58}$; $b_3=\beta_3+\beta_{57}$;
$b_4=\beta_4+\beta_{56}$; $b_5=\beta_5+\beta_{46}+\beta_{37}+\beta_{28}$; $b_6=\beta_6+\beta_{45}$;
$b_7=\beta_7+\beta_{35}$; $b_8=\beta_8+\beta_{25}$; $b_{12}=\beta_{12}+\beta_{38}+\beta_{47}$;
$b_{13}=\beta_{13}+\beta_{26}+\beta_{48}$; $b_{14}=\beta_{14}+\beta_{27}+\beta_{38}$; $b_{23}=\beta_{23}+\beta_{16}+\beta_{78}$;
$b_{24}=\beta_{24}+\beta_{17}+\beta_{68}$; $b_{34}=\beta_{34}+\beta_{18}+\beta_{67}$; $b_{15}=\beta_{15}$.

Depending on interaction significance, one experimental design may be used in different cases. Thus, a design of experiments with eight design points-trials may be used [23]:

- in the case of three factors, to calculate all main effects and all interactions (*FUFE*);
- in the case of four factors, to calculate main effects and two-factor interactions between three out of all in all four factors, hereby neglecting all other interactions;
- in the case of five factors, to calculate main effects and two two-factor interactions;
- in the case of six factors, to calculate main effects and one two-factor interaction;
- in the case of seven factors, to calculate main effects, neglecting all interactions.

Now consider a fractional replica of type $2^{15\text{-}11}$, which is the 1/2048-replica of a *FUFE*. It is pointless in this case to write down all aliased/confounded estimates as their number is enormous. As an example, linear effects are aliased/confounded with 105 even interactions. The design matrix of $2^{15\text{-}11}$ is shown in Table 2.96.

When forming the *design matrix* of an experiment it is transformed into an *operational matrix* by replacing coded values with associated real-actual, dimensional values. The experiment is done based on an operational matrix. When we obtain experimental values or responses, we again refer to the design matrix, which is then completed into *arithmetic matrix*, by addition of columns associated with interactions that are of interest. Furthermore, regression coefficients are calculated by the method of least squares as a special method of regression analysis. Linear regression coefficients are calculated by these formulas:

$$b_i=\frac{\sum\limits_{1}^{N}X_{iu}\times\bar{y}_u}{\sum\limits_{1}^{N}X_{iu}^2}=\frac{\sum\limits_{1}^{N}X_{iu}\times\bar{y}_u}{N} \tag{2.62}$$

Table 2.96 Fractional factorial design 2^{15-11}

No. of trials	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}
1	−	−	−	−	+	+	+	+	+	+	−	−	−	−	+
2	+	−	−	−	−	−	−	+	+	+	+	+	+	−	−
3	−	+	−	−	−	+	+	−	−	+	+	+	−	+	−
4	+	+	−	−	+	−	−	−	−	+	−	−	+	+	+
5	−	−	+	−	+	−	+	−	+	−	+	−	+	+	−
6	+	−	+	−	−	+	−	−	+	−	−	+	−	+	+
7	−	+	+	−	−	−	+	+	−	−	−	+	+	−	+
8	+	+	+	−	+	+	−	+	−	−	+	−	−	−	−
9	−	−	−	+	+	+	−	+	−	−	−	+	+	+	−
10	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+
11	+	+	−	+	+	−	+	−	+	−	−	+	−	−	−
12	−	−	+	+	+	−	−	−	−	+	+	+	−	−	+
13	+	−	+	+	−	+	+	−	−	+	−	−	+	−	−
14	−	+	+	+	−	−	−	+	+	+	−	−	−	+	−
15	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
16	+	−	−	+	−	−	+	+	−	−	+	−	−	+	+

where:
X_{iu} is coded X_i factor value in the u-th design point-trial;
$\bar{y}_u$ is response average in the u-th design point-trial;
N is total number of design points-trials in design matrix;
u is current number of design points-trials.

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The value of the free member in a regression equation (b_0) is determined from the relation:

$$b_0 = \frac{\sum_{1}^{N} \bar{y}_u}{N} \tag{2.63}$$

Regression coefficients of two-factors interactions are determined thus:

$$b_{ij} = \frac{\sum_{1}^{N} X_{iu} X_{ju} \bar{y}_u}{\sum_{1}^{N} X_{iu}^2} = \frac{\sum_{1}^{N} X_{iu} X_{ju} \bar{y}_u}{N} \tag{2.64}$$

After obtaining regression coefficient values, both their *statistical significance* and *lack of fit* of the obtained regression model are checked.

2.3.1.1 Yates Method

Section 1.5 dealt with the analysis of variance method, which may easily be applied for *FUFE* analysis of results. Hereby one should take care to transform the *FUFE* design arithmetic matrix into the table, which is required by the analysis of variance notation. One should also keep in mind the difference in processing designs with

and without replications of design points-trials. The classical procedure of processing analysis of variance outcomes becomes much more complex with an increase in the number of factors. To facilitate a routine processing of *FUFE* designs with a large number of factors, *Yates* [27, 28] has developed a simple procedure of presenting and processing outcomes. To simplify matters these signs are introduced: factors are labelled by Latin capitals, design points-trials with all its factors in lower level (1), and other design points-trials by small letters for factors in the upper levels. As example, for 2^3 *FUFE*, symbols for experimental conditions are shown in Table 2.97.

Table 2.97 FUFE 2^3 symbols

Trial symbols	Factor symbols		
	A	B	C
(1)	−	−	−
a	+	−	−
b	−	+	−
ab	+	+	−
c	−	−	+
ac	+	−	+
bc	−	+	+
abc	+	+	+

In accordance with former notation, symbols in columns denote variation levels of associated factors A, B and C.

A small letter in a design point-trial sign means that the factor it represents in a corresponding design point-trial is set at a higher level, while lack of small letters means that the associated factors are in the lower level. The sign (1) means that all factors are in the lower level; a design point-trial marked by (a) indicates that factor A is in the upper and factor B and C in the lower level, etc.

In accord with calculations by the least square method, the factor A affect is the difference of average design point-trials or response values in upper and lower levels:

$$A = 1/4(a + ab + ac + abc) - 1/4((1) + b + c + bc) \tag{2.65}$$

By treating (1), a, b and c as algebraic expressions we get:

$$A=1/4(a-1)(b+1)(c+1) \tag{2.66}$$

By analogy we obtain:

$$\begin{aligned} A&=1/4(a-1)(b+1)(c+1)\\ B&=1/4(a+1)(b-1)(c+1)\\ C&=1/4(a+1)(b+1)(c-1)\\ AB&=1/4(a-1)(b-1)(c+1) \end{aligned} \tag{2.67}$$

$AC=1/4(a-1)(b+1)(c-1)$
$BC=1/4(a+1)(b-1)(c-1)$
$ABC=1/4(a-1)(b-1)(c-1)$

The Yates algorithm is easily generalized for any 2^k *FUFE*. For k factor A, B, C, ..., Q, each on two levels, the associated expressions are:

$$\begin{aligned} A&=(1/2)^{k-1}(a-1)(b+1)(c+1)\ldots(q+1)\\ AB&=(1/2)^{k-1}(a-1)(b-1)(c+1)\ldots(q+1)\\ &\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\\ ABC\ldots Q&=(1/2)^{k-1}(a-1)(b-1)(c-1)\ldots(q-1)\end{aligned} \tag{2.68}$$

The equation for calculating effect A in its developed form may be written as:

$$4A=-1+a-b+ab-c+ac-bc+abc \tag{2.69}$$

The structure of the equation for determining an effect and sequence of design points-trials is the same, so that an effect may be directly determined from design point-trial or response outcomes by adding associated signs:

$$-\ +\ -\ +\ -\ +\ -\ +$$

Other effects and interactions may be expressed in a completely identical way. Table 2.98 shows *Yates* notation and processing for 2^2, 2^3, 2^4, 2^5, *FUFE*. The given procedure for calculating factor and interaction effects, as well as a specific aspect of two-level designs facilitates a rather simple method of processing results. As the number of degrees of freedom of factor and interaction effects is one, the sum of squares is identical to the variance estimate. Since factor effects are obtained from design point-trial or response outcome differences in upper and lower levels, and the experimental design is done in only two levels, it is possible to calculate the relationship between the sum of squares, or an estimate on variance and calculated effects:

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$$\textbf{Sum of squares } SS=2^{k-2}\,(\text{effect})^2 \tag{2.70}$$

Hence, based on calculated effects of individual factors and interactions from relation (2.70), we obtain a variance estimate, which is further analyzed by the analysis of variance method. Here, one should remember once again the difference between two cases in estimating residual variance:

- full factorial designs with no trial replications,
- full factorial designs with trial replications.

One can often find tabular presentation of *FRFE* in Anglo-Saxon reference literature. Let us keep here the Yates notation for seven factors A, B, C, D, E, F and G, which are all varied on two levels. According to *FUFE* such a design includes $2^7=128$ design points-trials that are given geometrically in Table 2.99. Shaded cells in the same table give a half-replica with $2^{7-1}=64$ design points-trials that belong to a 2^{7-1} fractional factorial experiment. Associated geometric interpretations of a 1/4-replica and of a 1/8-replica are shown in Tables 2.100 and 2.101.

Table 2.102 offers a researcher several very useful two-level fractional factor designs with effects that can be estimated (under assumptions that three-factor and multifactor interactions are negligible). The design of experiments matrix consist of trials that are given for each *FRFE* but in a completely random sequence.

Table 2.98 Main effects and interactions for $2^2; 2^3; 2^4; 2^5$ FUFE Yates notation

	T	A	B	AB	C	AC	BC	ABC	D	AD	BD	ABD	CD	ACD	BCD	ABCD	E	AE	BE	ABE	CE	ACE	BCE	ABCE	DE	ADE	BDE	ABDE	CDE	ACDE	BCDE	ABCDE
(1)	+	−	−	+	−	+	+	−	−	+	+	−	+	−	−	+	−	+	+	−	+	−	−	+	+	−	−	+	−	+	+	−
a	+	+	−	−	−	−	+	+	−	−	+	+	+	+	−	−	−	−	+	+	+	+	−	−	+	+	−	−	−	−	+	+
b	+	−	+	−	−	+	−	+	−	+	−	+	+	−	+	−	−	+	−	+	+	−	+	−	+	−	+	−	+	−	−	+
Ab	+	+	+	+	−	−	−	−	−	−	−	−	+	+	+	+	−	−	−	−	+	+	+	+	+	+	+	+	−	−	−	−
c	+	−	−	+	+	−	−	+	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	+	−	−	+	+	−	−	+
Ac	+	+	−	−	+	+	−	−	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	+	+	−	−	+	+	−	−
Bc	+	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	+	−	+	−	+	−	+	−
Abc	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−	+	+	+	+	+	+	+	+
d	+	−	−	+	−	+	+	−	+	−	−	+	−	+	+	−	−	+	+	−	+	−	−	+	−	+	+	−	+	−	−	+
Ad	+	+	−	−	−	−	+	+	+	+	−	−	−	−	+	+	−	−	+	+	+	+	−	−	−	−	+	+	+	+	−	−
Bd	+	−	+	−	−	+	−	+	+	−	+	−	−	+	−	+	−	+	−	+	+	−	+	−	−	+	−	+	+	−	+	−
Abd	+	+	+	+	−	−	−	−	+	+	+	+	−	−	−	−	−	−	−	−	+	−	+	+	−	−	−	−	+	+	+	+
Cd	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−
Acd	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+
Bcd	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+
Abcd	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−
e	+	−	−	+	−	+	+	−	−	+	+	−	+	−	−	+	+	−	−	+	−	+	+	−	−	+	+	−	+	−	−	+
Ae	+	+	−	−	−	−	+	+	−	−	+	+	+	+	−	−	+	+	−	−	−	−	+	+	−	−	+	+	+	+	−	−
Be	+	−	+	−	−	+	−	+	−	+	−	+	+	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+	+	−	+	−
Abe	+	+	+	+	−	−	−	−	−	−	−	−	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−	+	+	+	+
Ce	+	−	−	+	+	−	−	+	−	+	+	−	−	+	+	−	+	−	−	+	+	−	−	+	−	+	+	−	−	+	+	−
Ace	+	+	−	−	+	+	−	−	−	−	+	+	−	−	+	+	+	+	−	−	+	+	−	−	−	−	+	+	−	−	+	+
Bce	+	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+	+	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+
Abce	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−
De	+	−	−	+	−	+	+	−	+	−	−	+	−	+	+	−	+	−	−	+	−	+	+	−	+	−	−	+	−	+	+	−
Ade	+	+	−	−	−	−	+	+	+	+	−	−	−	−	+	+	+	+	−	−	−	−	+	+	+	+	−	−	−	−	+	+
Bde	+	−	+	−	−	+	−	+	+	−	+	−	−	+	−	+	+	−	+	−	−	+	−	+	+	−	+	−	−	+	−	+
Abde	+	+	+	+	−	−	−	−	+	+	+	+	−	−	−	−	+	+	+	+	−	−	−	−	+	+	+	+	−	−	−	−
Cde	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+
Acde	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−
Bcde	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−
Abcde	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+

Table 2.99 Fractional factorial design 2^{7-1}

			A(-)								A(+)							
			B(-)				B(+)				B(-)				B(+)			
			C(-)		C(+)		C(-)		C(+)		C(-)		C(+)		C(-)		C(+)	
			D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)
	F	G(-)		■		■			■			■		■				
E	(-)	G(+)	■		■			■			■				■			■
(-)	F	G(-)	■		■			■			■				■			■
	(+)	G(+)		■		■			■			■		■				
	F	G(-)	■		■			■			■				■			■
E	(-)	G(+)		■		■			■			■		■				
(+)	F	G(-)		■		■			■			■		■				
	(+)	G(+)	■		■			■			■				■			■

Table 2.100 Fractional factorial design 2^{7-2}

			A(-)								A(+)							
			B(-)				B(+)				B(-)				B(+)			
			C(-)		C(+)		C(-)		C(+)		C(-)		C(+)		C(-)		C(+)	
			D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)
	F	G(-)	■					■		■				■			■	
E	(-)	G(+)		■			■		■				■			■		■
(-)	F	G(-)			■			■				■			■			
	(+)	G(+)				■	■				■			■		■		
	F	G(-)			■			■				■			■			
E	(-)	G(+)				■	■						■			■		■
(+)	F	G(-)		■					■	■								■
	(+)	G(+)	■						■							■		

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Table 2.101 Fractional factorial design 2^{7-3}

			A(-)								A(+)							
			B(-)				B(+)				B(-)				B(+)			
			C(-)		C(+)		C(-)		C(+)		C(-)		C(+)		C(-)		C(+)	
			D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)	D(-)	D(+)
	F	G(-)																
E	(-)	G(+)																
(-)	F	G(-)																
	(+)	G(+)																
	F	G(-)																
E	(-)	G(+)																
(+)	F	G(-)																
	(+)	G(+)																

Example 2.26 [29]

In Example 2.12, the method of random balance, factors have been selected by the effects of their significance on dynamic viscosity of uncured composite rocket propellant. The screened-out factors are: X_3 mixing speed; X_5 time after addition of AP and X_8 vacuum in vertical planetary mixer. Since insufficient vacuum in a mixer causes bubbles to appear in the cured propellant, the value of this factor is fixed at the most convenient one. For the other two factors a design of basic experiment has been done according to a *FUFE* matrix, as shown in Table 2.103, and aimed at obtaining the mathematical model of viscosity change.

Table 2.102 Fractional factorial designs

Design	Trials	Effects
Design 1: 2^{3-1}	(1)	T
3 factors (k=3)	ac	A-BC
1/2 replica (p=1)	bc	B-AC
4 trials (n=4)	ab	-C+AB
	(1)	T
	ad	A
Design 2: 2^{4-1}	bd	B
4 factors (k=4)	ab	AB+CD
1/2 replica (p=1)	cd	C
8 trials (n=8)	ac	AC+BD
	bc	BC+AD
	abcd	D
	(1)	T
	ae	A
	be	B
	ab	AB
	ce	C
	ac	AC
Design 3: 2^{5-1}	bc	BC
5 factors (k=5)	abce	-DE
1/2 replica (p=1)	de	D
16 trials (n=16)	ad	AD
	bd	BD
	abde	-CE
	cd	CD
	acde	-BE
	bcde	-AE
	abed	-E

* – used to estimate experimental error

Design	Trials	Effects
	(1)	T
	ad	A-DE
Design 4: 2^{5-2}	bde	B-CE
5 factors (k=5)	abe	AB+CD
1/4 replica (p=2)	cde	C-BE
8 trials (n=8)	ace	AC+BD
	bc	-E+BC+AD
	abcd	D-AE
	(1)	T
	ae	A
	bef	B
	abf	AB+CE
	cef	C
	acf	AC+BE
Design 5: 2^{6-2}	bc	BC+AE+DF
6 factors (k=6)	abce	E
1/4 replica (p=2)	df	D
16 trials (n=16)	adef	AD+EF
	bde	BD+CF
	abd	*
	cde	CD+BF
	acd	*
	bcdf	F
	abcdef	AF+DE
	(1)	T
	adf	A-DE-CF
Design 6: 2^{6-3}	bde	B-CE-DF
6 factors(k=6)	abef	AB+CD+EF
1/8 replica (p=3)	cdef	C-AF-BE
8 trials (n=8)	ace	-F+AC+BD
	bcf	-E+AD+BC
	abed	D-AE-BF

Design	Trials	Effects
	(1)	T
	aeg	A
	befg	B
	abf	AB+CE+DG
	cef	C
	acfg	AC+BE+FG
Design 7:2^{7-3}	bcg	BC+AE+DF
7 factors (k=7)	abce	E
1/8 replica (p=3)	dfg	D
16 trials (n=16)	adef	AD+EF+BG
	bde	BD+CF+AG
	abdg	G
	cdeg	CD+BF+EG
	acd	*
	bcdf	F
	abcdefg	AF+DE+CG
	(1)	T
	aegh	A
	befg	B
	abfh	AB+CE+DG+FH
	cefh	C
	acfg	AC+BE+FG+DH
Design 8:2^{8-4}	bcgh	BC+AE+DF+GH
8 factors (k=8)	abce	E
1/16 replica (p=4)	dfgh	D
16 trials (n=16)	adef	AD+EF+BG+CH
	bdeh	BD+AG+CF+EH
	abdg	G
	cdeg	CD+AH+BF+EG
	acdh	H
	bcdf	F
	abcdefgh	AF+DE+CG+BH

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Table 2.103 Full factorial experiment 2^2

Name	Mixing speed x_1 min^{-1}		Time x_2 min								
Basic level	60		95								
Variation interval	20		85								
Upper level	80		180								
Lower level	40		10								
Trials	**Design matrix**		**Oper. matrix**		**Response-viscosity p**						
	X_1	X_2	x_1	x_2	y_{u1}	y_{u2}	y_{u3}	y_{u4}	y_{u5}	y_{u6}	$\bar{y}_u$
1	−	−	40	10	1182.4	1139.2	1136.5	1209.4	1134.6	1159.2	1160.2
2	+	−	80	10	622.4	660.8	631.8	602.1	668.6	645.6	638.5
3	−	+	40	180	683.2	624.0	682.6	699.5	565.4	611.2	644.3
4	+	+	80	180	496.0	486.2	495.6	513.6	450.9	467.2	484.9
Sum											2927.54

Regression coefficients are determined in accordance with relations (2.62) – (2.64):

$$b_0 = \frac{\sum_1^N \bar{y}_u}{N} = \frac{2927.54}{4} = 731.98$$

$$b_1 = \frac{\sum_1^N X_{1u} \times y_u}{N} = \frac{-1160.2+638.5-644.3+484.9}{4} = -170.28$$

$$b_2 = \frac{\sum_1^N X_{2u} \times y_u}{N} = \frac{-1160.2-638.5+644.3+484.9}{4} = -167.38$$

$$b_{12} = \frac{\sum_1^N X_{1u} \times X_{2u} \times y_u}{N} = \frac{1160.2-638.5-644.3+484.9}{4} = 90.58$$

The mathematical model or linear regression equation has the form:

$$\hat{y} = 731.98 - 170.28X_1 - 167.38X_2 + 90.58X_1X_2$$

Since the relation between actual and coded factors is given by expressions:

$$X_1 = \frac{x_1-60}{20}; \quad X_2 = \frac{x_2-95}{85};$$

the regression equation with actual factors is:

$$\hat{y} = 731.98 - 170.28\frac{x_1-60}{20} - 167.38\frac{x_2-95}{85} + 90.58\frac{x_1-60}{20}\frac{x_2-95}{85}$$

Example 2.27 [30]

It is necessary to do mathematical modeling of effects of X_1 dyeing time, X_2 dye concentration and X_3 temperature of dyeing on efficiency of dyeing. For this, the *FUFE* design from Table 2.93 has been used for the three mentioned factors. Formula (2.59) has been used in coding the factors, with basic level and variation intervals being taken into account:

$$X_1 = \frac{x_1 - 10}{5}; \quad X_2 = \frac{x_2 - 1.0}{0.5}; \quad X_3 = \frac{x_3 - 85}{10};$$

The design matrix with operational matrix and outcomes of design points-trials is given in Table 2.104. Note that design points-trials have been replicated so that the table gives response means.

Table 2.104 Full factorial experiment 2^3

No. trials	Design matrix								Operational-matrix			Response $\sum \bar{y}_u$
	X_0	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3	$X_1X_2X_3$	x_1 min	x_2%	x_3°C	
1	+	−	−	−	+	+	+	−	5	0.5	75	81.08
2	+	+	−	−	−	−	+	+	15	0.5	75	85.65
3	+	−	+	−	−	+	−	+	5	1.5	75	82.27
4	+	+	+	−	+	−	−	−	15	1.5	75	90.40
5	+	−	−	+	+	−	−	+	5	0.5	95	84.95
6	+	+	−	+	−	+	−	−	15	0.5	95	89.95
7	+	−	+	+	−	−	+	−	5	1.5	95	85.25
8	+	+	+	+	+	+	+	+	15	1.5	95	88.25

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Regression coefficients of linear regression have these values:

b_0=85.98; b_1=2.60; b_2=0.54; b_3=1.13;
b_{12}=0.20; b_{13}=-0.59; b_{23}=-0.92; b_{123}=-0.70

and the linear regression has the form:

$$\widehat{y} = 85.98 + 2.60X_1 + 0.54X_2 + 1.13X_3 + 0.20X_1X_2 - 0.59X_1X_3 - 0.92X_2X_3 - 0.70X_1X_2X_3$$

Example 2.28 [21]

This example analyzes lab research of yields in a nitration process, which gives the basic product for medicine and dye industries. Three factors assumed to have effects on yield in the nitration process have been researched:

1. A nitric acid dosing time, h;
2. B mixing time, h;
3. C factor of mixing remnants from previous batch.

These factor levels have been used in the experiment:

Factors	Lower level	Upper level
A	2	7
B	0.5	4
C	with no mixture remnants	with mixture remnants

Do the outcome analysis by classical and *Yates* processing. The measurement results are in Table 2.105.

Table 2.105 Full factorial experiment 2^3

No.	Yates	Design matrix								Operational matrix			Response
		T	A	B	AB	C	AC	BC	ABC	A	B	C	y_u
1	(1)	+	−	−	+	−	+	+	−	2	0.5	WITHOUT	87.2
2	a	+	+	−	−	−	−	+	+	7	0.5	WITHOUT	88.4
3	b	+	−	+	−	−	+	−	+	2	4	WITHOUT	82.0
4	ab	+	+	+	+	−	−	−	−	7	4	WITHOUT	83.0
5	c	+	−	−	+	+	−	−	+	2	0.5	WITH	86.7
6	ac	+	+	−	−	+	+	−	−	7	0.5	WITH	89.2
7	bc	+	−	+	−	+	−	+	−	2	4	WITH	83.4
8	abc	+	+	+	+	+	+	+	+	7	4	WITH	83.7

According to Eq. (2.67) we get:
A=1/4(a+ab+ac+abc)-1/4((1)+b+c+bc)=(88.4+83.0+89.2+83.7)/
4-(87.2+82.0+86.7+83.4)/4
A=1.25
B=1/4(b+ab+bc+abc)-1/4((1)+a+c+ac)=(82.0+83.0+83.4+83.7)/
4-(87.2+88.4+86.7+89.2)/4
B=-4.85
C=1/4(c+ac+bc+abc)-1/4((1)+a+b+ab)=(86.7+89.2+83.4+83.7)/
4-(87.2+88.4+82.0+83.0)/4
C=0.60
AB=1/4((1)+ab+c+abc)-1/4(a+b+ac+bc)=(87.2+83.0+86.7+83.7)/
4-(88.4+82.0+89.2+83.4)/4
AB=-0.60
AC=1/4((1)+b+ac+abc)-1/4(a+ab+c+bc)=(87.2+82.0+89.2+83.7)/
4-(88.4+83.0+86.7+83.4)/4
AC=0.15
BC=1/4((1)+a+bc+abc)-1/4(b+ab+c+ac)=(87.2+88.4+83.4+83.7)/
4-(82.0+83.0+86.7+89.2)/4
BC=0.45
ABC=1/4(a+b+c+abc)-1/4((1)+ab+ac+bc)=(88.4+82.0+86.7+83.7)/
4-(87.2+83.0+89.2+83.4)/4
ABC=-0.50;
T=((1)+a+b+ab+c+ac+bc+abc)=683.60.

Since the effects are doubled in comparison with regression coefficients, it follows:

$b_0=T/8=85.45$; $b_A=A/2=0.63$; $b_B=B/2=-2.43$; $b_C=C/2=0.30$;
$b_{AB}=AB/2=-0.30$; $b_{AC}=AC/2=0.075$; $b_{BC}=BC/2=0.225$; $b_{ABC}=ABC/2=-0.25$.

To demonstrate the relation between effects and estimates of associated variances, we will transform Table 2.105 into Table 2.106.

Table 2.106 Analysis of variance

Factors	C(-)		C(+)	
	A(-)	A(+)	A(-)	A(+)
B(-)	(1) 87.2	a 88.4	c 86.7	ac 89.2
B(+)	b 82.0	ab 83.0	bc 83.4	abc 83.7

Variance estimates based on f=1 degrees of freedom are calculated from effects from Eq. (2.70):

$MS_A=2^{k-2} \times A^2=2^{3-2} \times 1.25^2=3.125$; $MS_B=2(-4.85)^2=47.045$;
$MS_C=2 \times 0.6^2=0.720$; $MS_{AB}=2(-0.6)^2=0.720$; $MS_{AC}=2 \times 0.15^2=0.045$;
$MS_{BC}=2 \times 0.45^2=0.405$; $MS_{ABC}=2(-0.5)^2=0.500$.

Do not forget that the experiment has been done with no replication of trial, so that residual variance must be determined based on the interaction variances. To check whether all interactions may be replaced or some variance interactions not be included into the residual variance, (as its effect is important and different from others) use the *Bartlett* criterion, as shown in Sect. 1.5. By comparing the analysis of variance outcomes from Problem 1.34 with values obtained here, it is evident that completely identical outcomes are in question.

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Example 2.29 [21]

In lab studies of isatin yield, conditions of the technological procedure in producing this product from isonitrozoacetylamine have been tested. The effects of three process factors have been assessed.

	Lower level	Upper level
A concentration of basic raw material, %	87	93
B duration of reaction, min	15	30
C temperature of reaction, °C	60	70

Based on previous testing of the research subject, the design of the full factorial experiment 2^3 with one replication to determine experimental error has been chosen. To eliminate the influence of systematic error in doing the experiment, the sequence of doing design point-trials, in accord with theory of design of experiments, has been completely random. The outcomes are given in Table 2.107.

Table 2.107 Full factorial experiment 2^3

No.	Yates	Design matrix								Operational matrix			Response		
		T	A	B	AB	C	AC	BC	ABC	A	B	C	y_{u1}	y_{u2}	$\sum y_u$
1	(1)	+	−	−	+	−	+	+	−	87	15	60	6.08	6.31	12.39
2	a	+	+	−	−	−	−	+	+	93	15	60	6.04	6.09	12.13
3	b	+	−	+	−	−	+	−	+	87	30	60	6.53	6.12	12.65
4	ab	+	+	+	+	−	−	−	−	93	30	60	6.43	6.36	12.79
5	c	+	−	−	+	+	−	−	+	87	15	70	6.79	6.77	13.56
6	ac	+	+	−	−	+	+	−	−	93	15	70	6.68	6.38	13.06
7	bc	+	−	+	−	+	−	+	−	87	30	70	6.73	6.49	13.22
8	abc	+	+	+	+	+	+	+	+	93	30	70	6.08	6.23	12.31

According to Eq. (2.67) we get:

A=-0.191; B=-0.021; C=0.274; AB=-0.001;
AC=-0.161; BC=-0.251; ABC=-0.101; T=51.055.

The sum of squares of effects and interactions or mean square (f=1), is obtained from Eq. (2.70):

$MS_A=2^{k-1}n(Effect)^2 = 2 \times 2(-0.191)^2=0.146$

$MS_B=0.002$; $MS_C=0.300$; $MS_{AB}=0.000$; $MS_{AC}=0.104$; $MS_{BC}=0.253$; $MS_{ABC}=0.041$;

$$SS_E = \frac{\sum_{1}^{N}\sum_{1}^{n}\left(y_{ui}-\bar{y}_u\right)^2}{n-1} = 0.200; \qquad MS_E = SS_E/N = 0.200/8 = 0.025.$$

Note that in preliminary calculations the sum of replicated design points-trials is taken as the response, and thus the number of replicated design points n is introduced Eq. (2.70). As there exists replication of trials, it is evident that the error sum of squares is calculated in accord with analysis of variance methodology. To enable comparison of such variance determination with classical analysis of variance, it is necessary to transform Table 2.107 into Table 2.108.

Table 2.108 Analysis of variance

Factors	C(-)		C(+)	
	B(-)	B(+)	B(-)	B(+)
	6.08	6.53	6.79	6.73
A(-)	(1)	b	c	bc
	6.31	6.12	6.77	6.49
	6.04	6.43	6.68	6.08
A(+)	a	ab	ac	abc
	6.09	6.36	6.38	6.23

According to calculation of analysis of variance from Sect. 1.5 we get:

$$\sum\sum\sum\sum y_{ijkl}^2=652.70; \qquad y_{\bullet\bullet\bullet} = 102.11 \qquad y_{\bullet\bullet\bullet}^2 = 12426.45;$$

$$\sum_i y_{i\bullet\bullet\bullet}^2=51.82^2+50.29^2=5214.40$$

$$\sum_j y_{\bullet j\bullet\bullet}^2=(24.52+25.44)^2+(26.62+25.53)^2=5215.62$$

$$\sum_k y_{\bullet\bullet k\bullet}^2=(24.52+26.62)^2+(25.44+25.53)^2=5213.24$$

$$\sum_i\sum_j y_{ij\bullet\bullet}^2=(12.39+12.65)^2+(13.56+13.22)^2+(12.13+12.79)^2+(13.06+12.31)^2$$
$$=2608.82$$

$$\sum_i\sum_k y_{i\bullet k\bullet}^2=(12.39+13.56)^2+(12.13+13.06)^2+(12.65+13.22)^2+(12.79+12.31)^2$$
$$=2607.21$$

$$\sum_j\sum_k y_{\bullet jk\bullet}^2=(12.39+12.13)^2+(12.65+12.79)^2+(13.56+13.06)^2+(13.22+12.31)^2$$
$$=2608.82$$

$$\sum_i\sum_j\sum_k y_{ijk\bullet}^2=12.39^2+12.65^2+13.56^2+13.22^2+12.13^2+12.79^2+13.06^2+12.31^2$$
$$=1305.00$$

$SS_T=652.70-10426.45/16=1.045$
$SS_C=5215.62/8-651.65=0.300$
$SS_R=5214.40/8-651.65=0.146$
$SS_L=5213.24/8-651.65=0.002$
$SS_{CR}=2608.82/4-5214.40/8-5215.62/8+651.65=0.104$
$SS_{CL}=2608.82/4-5215.62/8-5213.24/8+651.65=0.248$
$SS_{RL}=2607.21/4-5214.40/8-5213.24/8+651.65=0.000$
$SS_E=652.70-1305.00/2=0.200$
$SS_{CRL}=1.045-0.300-0.146-0.002-0.104-0.248-0.200=0.04$

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The completed table of analysis of variance is given in solutions of Problem 1.26

Example 2.30 [21]

A research expansion with another factor has been done in the previous example. Hence, the effects of these factors have been analyzed in this research:

	Lower level	Upper level
A concentration of basic raw material, %	87	93
B duration of reaction, min	15	30
C quantity of basic raw material, ml	35	45
D temperature of reaction, °C	60	70

The experiment has been done through the matrix of full factorial experiment 2^4, as shown in Table 2.109. Each trial has been done only once, with no replications. The sequence of doing trials has been completely random.

Table 2.109 Full factorial experiment 2^4

No. trials	Yates	Design matrix																Operational matrix				Response
		T	A	B	AB	C	AC	BC	ABC	D	AD	BD	ABD	CD	ACD	BCD	ABCD	A	B	C	D	y_u
1	(1)	+	−	−	+	−	+	+	−	−	+	+	−	+	−	−	+	87	15	35	60	6.08
2	a	+	+	−	−	−	−	+	+	−	−	+	+	+	+	−	−	93	15	35	60	6.04
3	b	+	−	+	−	−	+	−	+	−	+	−	+	+	−	+	−	87	30	35	60	6.53
4	ab	+	+	+	+	−	−	−	−	−	−	−	−	+	+	+	+	93	30	35	60	6.43
5	c	+	−	−	+	+	−	−	+	−	+	+	−	−	+	+	−	87	15	45	60	6.31
6	ac	+	+	−	−	+	+	−	−	−	−	+	+	−	−	+	+	93	15	45	60	6.09
7	bc	+	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+	87	30	45	60	6.12
8	abc	+	+	+	+	+	+	+	+	−	−	−	−	−	−	−	−	93	30	45	60	6.36
9	d	+	−	−	+	−	+	+	−	+	−	−	+	−	+	+	−	87	15	35	70	6.79
10	ad	+	+	−	−	−	−	+	+	+	+	−	−	−	−	+	+	93	15	35	70	6.68
11	bd	+	−	+	−	−	+	−	+	+	−	+	−	−	+	−	+	87	30	35	70	6.73
12	abd	+	+	+	+	−	−	−	−	+	+	+	+	−	−	−	−	93	30	35	70	6.08
13	cd	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	87	15	45	70	6.77
14	acd	+	+	−	−	+	+	−	−	+	+	−	−	+	+	−	−	93	15	45	70	6.38
15	bcd	+	−	+	−	+	−	+	−	+	−	+	−	+	−	+	−	87	30	45	70	6.49
16	abcd	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	93	30	45	70	6.23

According to Eq. (2.67) we get:

A=-0.191; B=-0.021; AB=-0.001; C=-0.076; AC=0.034;
BC=-0.066; ABC=0.149; D=0.274; AD=-0.161; BD=-0.251;
ABD=-0.101; CD=-0.026; ACD=-0.006; BCD=0.124; ABCD=0.019

Associated sums of squares are:

$SS_A=MS_A=2^{k-2}A^2=2^2(-0.191)^2=0.1463$;
$MS_B=0.0018$; $MS_C=0.0233$; $MS_D=0.2998$; $MS_{AB}=0.0000$;
$MS_{AC}=0.0046$; $MS_{AD}=0.1040$; $MS_{BC}=0.0176$; $MS_{BD}=0.2525$;
$MS_{CD}=0.0028$; $MS_{ABC}=0.0885$; $MS_{ABD}=0.0410$; $MS_{ACD}=0.0002$;
$MS_{BCD}=0.0613$; $MS_{ABCD}=0.0014$.

By transforming Table 2.109 into Table 2.110 we get analysis of variance:

Table 2.110 Analysis of variance

		D(-)		D(+)	
		C(-)	C(+)	C(-)	C(+)
A(-)	B(-)	(1) 6.08	c 6.31	d 6.79	cd 6.77
	B(+)	B 6.53	bc 6.12	bd 6.73	bcd 6.49
A(+)	B(-)	A 6.04	ac 6.09	ad 6.68	acd 6.38
	B(+)	Ab 6.43	abc 6.36	abd 6.08	abcd 6.23

The outcomes of analysis of variance are shown in Table 2.111.

Table 2.111 Results of analysis of variance

Sources of variation	f	MS	F	$F_{1;5;0.95}$
Concentration A	1	0.1463	0.76	6.61
Reaction time B	1	0.0018	0.01	6.61
Quantity of raw material C	1	0.0233	0.12	6.61
Temperature of reaction D	1	0.2998	1.56	6.61
AB	1	0.0000	0.00	6.61
AC	1	0.0046	0.02	6.61
AD	1	0.1040	0.54	6.61
BC	1	0.0176	0.09	6.61
BD	1	0.2525	1.31	6.61
CD	1	0.0028	0.01	6.61
ABC+ABD+ACD+BCD+ABCD	5	0.1924	–	–
Total	15	–	–	–

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Example 2.31 [25]

A full factorial experiment has been done in a pilot-plant. The research included refinement of a product by steam distillation. Five factors have been analyzed, each one at two levels: A concentration, B flow, C volume of solution, D mixing speed and E solvent and water ratio. Acidity of the product in each of 32 trials has been analyzed as the response. Outcomes in coded forms are shown in Table 2.112.

Data from Table 2.112 have been analyzed by the Yates technique and outcomes are given in Table 2.113. The interesting thing in relation to the former example is that the mechanical method, which does not require knowledge of Eq. (2.67) has been demonstrated. Column (1) is obtained by adding up the response data pairs to the column and then by subtracting the data. For example, 19=9+10, 14=8+6,...,11=5+6, 1=10-9, -2=6-8,...,1=6-5. As shown, differences are taken from the same data pairs but in this way: the second data minus the first, the fourth minus the third and so on to the column end. Column (2) is obtained from the first column in the same way. Column (3) from (2), (4) from (3) and (5) from (4). This calculation is evidently repeated k times for a full factorial experiment of 2^k. Column (5) gives

Table 2.112 Full factorial experiment 2^5

		A(-)				A(+)			
		D(-)		D(+)		D(-)		D(+)	
		E(-)	E(+)	E(-)	E(+)	E(-)	E(+)	E(-)	E(+)
B(-)	C(-)	9	3	11	8	10	9	13	7
	C(+)	3	5	7	7	5	6	10	7
B(+)	C(-)	8	4	9	8	6	6	16	6
	C(+)	6	4	7	5	10	10	13	6

total effects of the factors and interactions. Average effects are obtained by dividing the totals with N/2.

At the end, the last column represents the sum of squares of factors and interactions. This one can be obtained by dividing the square of values of column (5) (total effects) by the total number of trials $N=2^k$.

Table 2.113 Yates method 2^5

Trials	Response	(1)	(2)	(3)	(4)	(5)	Total effects	Average effects(5)/16	SS=MS $(5)^2/32$
(1)	9	19	33	57	143	244	T	–	–
a	10	14	24	86	101	36	16A	2.250	40.500
b	8	8	49	47	23	4	16B	0.250	0.500
ab	6	16	37	54	13	8	16AB	0.500	2.000
c	3	24	22	5	7	-22	16C	-1.375	15.125
ac	5	25	25	18	-3	10	16AC	0.625	3.125
bc	6	17	29	15	7	18	16BC	1.125	10.125
abc	10	20	25	-2	1	14	16ABC	0.875	6.125
d	11	12	-1	3	-21	36	16D	2.250	40.500
ad	13	10	6	4	-1	-4	16AD	-0.250	0.500
bd	9	11	9	1	7	-4	16BD	-0.250	0.500
abd	16	14	9	-4	3	8	16ABD	0.500	2.000
cd	7	15	8	-1	15	-10	16CD	-0.625	3.125
acd	10	14	7	8	3	-2	16ACD	-0.125	0.125
bcd	7	14	-3	1	3	-18	16BCD	-1.125	10.125
abcd	13	11	1	0	11	-14	16ABCD	-0.875	*6.125
e	3	1	-5	-9	29	-42	16E	-2.625	55.125
ae	9	-2	8	-12	7	-10	16AE	-0.625	3.125
be	4	2	1	3	13	-10	16BE	-0.625	3.125
abe	6	4	3	-4	-17	-6	16ABE	-0.375	1.125
ce	5	2	-2	7	1	20	16CE	1.250	12.500
ace	6	7	3	0	-5	-4	16ACE	-0.250	0.500

Table 2.113 (continued)

Trials	Response	(1)	(2)	(3)	(4)	(5)	Total effects	Average effects(5)/16	SS=MS $(5)^2/32$
bce	4	3	-1	-1	9	-12	16BCE	-0.750	4.500
abce	10	6	-3	4	-1	8	16ABCE	0.500	*2.000
de	8	6	-3	13	-3	-22	16DE	-1.375	15.125
ade	7	2	2	2	-7	-30	16ADE	-1.875	28.125
bde	8	1	5	5	-7	-6	16BDE	-0.375	1.125
abde	6	6	3	-2	5	-10	16ABDE	-0.625	*3.125
cde	7	-1	-4	5	-11	-4	16CDE	-0.250	0.500
acde	7	-2	5	-2	-7	12	16ACDE	0.750	*4.500
bcde	5	0	-1	9	-7	4	16BCDE	0.250	*0.500
abcde	6	1	1	2	-7	0	16ABCDE	0.000	*0.000
Total	244								

If in Table 2.113 we look at values of the sum of squares, we may, without testing them by Bartletts criterion, assume that the fourth- and fifth-order interactions are insignificant. That is, those sums of squares, marked by asterisks, may be used in analysis of variance in Table 2.114 as residual variance.

Table 2.114 Analysis of variance

Sources of variations	SS	f	MS	F	$F_{1;6;0.95}$
A	40.500	1	40.500	14.96	5.99
B	0.500	1	0.500	–	5.99
C	15.125	1	15.125	5.58	5.99
D	40.500	1	40.500	14.96	5.99
E	55.125	1	55.125	20.36	5.99
AB	2.000	1	–	–	–
AC	3.125	1	–	–	–
AD	0.500	1	–	–	–
AE	3.125	1	–	–	–
BC	10.125	1	–	–	–
BD	0.500	1	–	–	–
BE	3.125	1	–	–	–
CD	3.125	1	–	–	–
CE	12.500	1	12.500	4.62	5.99
DE	15.125	1	15.125	5.58	5.99
ABC	6.125	1	–	–	–
ABD	2.000	1	–	–	–
ABE	1.125	1	–	–	–
ACD	0.125	1	–	–	–

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Table 2.114 (continued)

Sources of variations	SS	f	MS	F	$F_{1;6;0.95}$
ACE	0.500	1	–	–	–
ADE	28.125	1	28.125	10.4	5.99
BCD	10.125	1	–	–	–
BCE	4.500	1	–	–	–
BDE	1.125	1	–	–	–
CDE	0.500	1	–	–	–
Residual	16.250	6	2.708	–	–
Total	275.500	31	–	–	–

Example 2.32 [25]

In developing a practical industrial fermentation process, we usually start with lab studies of micro-organism physiological requirements. Micro-organisms are cultivated in a liquid media and their growth depends on the substance being formed in that medium. Formation of the substance, according to previous research, depends on two components in the medium and two environmental factors: temperature and aeration. The first two factors are X_1 and X_2 and the other two X_3 and X_4. The experiment has been done by a full factorial type 2^4 with a single replication. Outcomes of experiment are shown in Table 2.115.

Table 2.115 Full factorial experiment 2^4

		X_1(-)		X_1(+)	
		X_2(-)	X_2(+)	X_2(-)	X_2(+)
	X_3	32.7	90.4	70.6	115.0
X_4	(-)	19.3	89.8	84.5	108.6
(-)	X_3	20.2	94.1	76.1	133.6
	(+)	29.9	96.5	73.3	131.6
	X_3	50.0	72.6	104.2	81.3
X_4	(-)	52.1	76.9	103.4	88.2
(+)	X_3	50.5	91.8	78.6	108.3
	(+)	49.1	86.9	74.1	108.3

The results of analysis of data by Yates methodology are shown in Table 2.116. Note that, here, the sums of replicate trials have been taken as response values. Therefore, when calculating, average effects are divided 2(N/2). The situation is similar when calculating sums of factor squares and interactions. Sum of squares are calculated by dividing squares of total effects by total number of data $N=n2^k$ (n-number of replicate trials). Residual sum of squares, as said before, has been determined according to the equation:

$$SS_E = \frac{\sum_{1}^{N}\sum_{1}^{n}\left(y_{ui}-\bar{y}_u\right)^2}{n-1} = 321.6$$

$$MS_E = \frac{SS_E}{N} = \frac{321.6}{16} = 20.1$$

Table 2.116 Yates method

Trials	Response	(1)	(2)	(3)	(4)	Average effects (4)/16	MS $(4)^2/32$	F	$F_{1;16;0.95}$
(1)	52.0	207.1	610.9	1239.6	2542.5	–	–	–	–
X_1	155.1	403.8	628.7	1302.9	536.9	33.56	9008.2	448.17	4.49
X_2	180.2	309.7	655.3	272.0	605.3	37.83	11449.6	569.63	4.49
X_1X_2	223.6	319.0	647.6	264.9	-185.1	-11.57	1070.7	53.27	4.49
X_3	102.1	199.5	146.5	206.0	10.1	0.63	3.2	0.16	4.49
X_1X_3	207.6	455.8	125.5	399.3	-103.9	-6.49	337.4	16.79	4.49
X_2X_3	149.5	252.3	173.9	-145.2	-300.7	-18.79	2825.6	140.58	4.49
$X_1X_2X_3$	169.5	395.3	91.0	-39.9	-16.3	-1.02	8.3	0.41	4.49
X_4	50.1	103.1	196.7	17.8	63.3	3.96	125.2	6.23	4.49
X_1X_4	149.4	43.4	9.3	-7.7	-7.1	-0.44	1.6	0.08	4.49
X_2X_4	190.6	105.5	256.3	-21.0	193.3	12.08	1167.7	58.09	4.49
$X_1X_2X_4$	265.2	20.0	143.0	-82.9	105.3	6.58	346.5	17.24	4.49
X_3X_4	99.6	99.3	-59.7	-187.4	-25.5	-1.59	20.3	1.01	4.49
$X_1X_3X_4$	152.7	74.6	-85.5	-113.3	-61.9	-3.87	119.7	5.96	4.49
$X_2X_3X_4$	178.7	53.1	-24.7	-25.8	74.1	4.63	171.6	8.54	4.49
$X_1X_2X_3X4$	216.6	37.9	-15.2	9.5	35.3	2.21	38.9	1.94	4.49
Residual	–	–	–	–	–	–	20.1	–	–
Total	–	–	–	–	–	–	–	–	–

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Example 2.33 [31]

This example refers to the use of fractional replica *FUFE* in testing adhesion of a thermoplastic polymer and fibre with inclusion of k=7 factors. Application of a fractional replica has proved to be specially useful since *FUFE* requires 2^7=128 trials and enormous time consumption. *FRFE* type 2^{7-3} with only 16 trials has been used. *FUFE* 2^4 has been used to construct the fractional replica. Here, the following generating ratios have been introduced: X_5=$X_1X_2X_4$; X_6=$X_2X_3X_4$; X_7=$X_1X_2X_3X_4$. Excluding all effects of triple and higher interactions, these aliased/confounded estimates of regression coefficients have been obtained:

$$b_1 = \beta_1 + \beta_{67};\quad b_2 = \beta_2;\quad b_3 = \beta_3 + \beta_{57};\quad b_4 = \beta_4;\quad b_5 = \beta_5 + \beta_{37};$$

$$b_6 = \beta_6 + \beta_{17};\quad b_7 = \beta_7 + \beta_{35} + \beta_{16};\quad b_{12} = \beta_{12} + \beta_{45};\quad b_{13} = \beta_{13} + \beta_{56};$$

$b_{14} = \beta_{14} + \beta_{25}$; $b_{23} = \beta_{23} + \beta_{46}$; $b_{24} = \beta_{24} + \beta_{36} + \beta_{15}$; $b_{34} = \beta_{34} + \beta_{26}$;

$b_{27} = \beta_{27}$; $b_{47} = \beta_{47}$;

Factors signs with variation intervals are shown in Table 2.117.

Table 2.117 Factors with variation intervals

Factors	Variation intervals			Δx
	–	0	+	
x_1 pressing pressure, kp/cm^2	5.0	12.5	20.0	7.5
x_2 thermal processing time, min	0.5	1.5	2.5	1.0
x_3 temperature, °C	140	155	170	15
x_4 pressing time, min	1	2	3	1
x_5 dibutylphthalate, %	0	0.5	1.0	0.5
x_6 tearing speed, m/min	80	140	200	60
x_7 fiber type	VISCOSE	–	KAPRON	–

The design matrix with the outcomes of experiment is shown in Table 2.118. Trials have been replicated 20 times due to the unreliable method of measuring adhesion.

Table 2.118 Fractional factorial experiment 2^{7-5}

No. trials	Design matrix								Operational matrix							Response
	X_0	X_1	X_2	X_3	X_4	X_5	X_6	X_7	x_1	x_2	x_3	x_4	x_5	x_6	x_7	$\bar{y}_u$
1	+	+	+	+	+	+	+	+	20	2.5	170	3	1.0	200	K	17.99
2	+	–	+	+	+	–	+	–	5	2.5	170	3	0	200	V	15.31
3	+	+	–	+	+	–	–	–	20	0.5	170	3	0	80	V	17.16
4	+	–	–	+	+	+	–	+	5	0.5	170	3	1.0	80	K	14.75
5	+	+	+	–	+	+	–	–	20	2.5	140	3	1.0	80	V	35.49
6	+	–	+	–	+	–	–	+	5	2.5	140	3	0	80	K	33.17
7	+	+	–	–	+	–	+	+	20	0.5	140	3	0	200	K	38.30
8	+	–	–	–	+	+	+	–	5	0.5	140	3	1.0	200	V	24.39
9	+	+	+	+	–	–	–	–	20	2.5	170	1	0	80	V	32.23
10	+	–	+	+	–	+	–	+	5	2.5	170	1	1.0	80	K	45.64
11	+	+	–	+	–	+	+	+	20	0.5	170	1	1.0	200	K	41.17
12	+	–	–	+	–	–	+	–	5	0.5	170	1	0	200	V	19.17
13	+	+	+	–	–	–	+	+	20	2.5	140	1	0	200	K	17.55
14	+	–	+	–	–	+	+	–	5	2.5	140	1	1.0	200	V	14.49
15	+	+	–	–	–	+	–	–	20	0.5	140	1	1.0	80	V	18.52
16	+	–	–	–	–	–	–	+	5	0.5	140	1	0	80	K	12.50

Based on the obtained experimental results, regression coefficients of linear regression have been determined by using Eqs. (2.62)–(2.64).

$$\widehat{Y}=24.86+2.44\,X_1+1.62\,X_2+0.56\,X_3-0.30\,X_4+3.14\,X_5-1.33\,X_6+2.77\,X_7$$

Example 2.34

To produce a textile material resistant to fire, these four qualitative factors have been tested:

Factors	Lower level	Upper level
1. A textile material	satin	monks
2. B textile treatment	treatment X	treatment Y
3. C washing conditions	before washing	after washing
4. D direction of testing	alongside	crosswise

The experiment was done by *FRFE* matrix of type 2^{4-1}. The response was the burnt-out part of the textile 25.4mm long. Determine the factor and interaction effects, since there is no point in determining regression coefficients as the factors are qualitative. To construct fractional 2^{4-1}, the design 2 from Table 2.102 was used. The sequence of trials in the design matrix has been completely random. Outcomes are shown in Table 2.119.

Table 2.119 Fractional replica 2^{4-1}

		A(-)		A(+)	
		B(-)	B(+)	B(-)	B(+)
C	D(-)	(1) 4.2	–	–	ab 2.9
(-)	D(+)	–	bd 5.0	ad 3.0	–
C	D(-)	–	bc 4.6	ac 2.8	–
(+)	D(+)	cd 4.0	–	–	abcd 2.3

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Table 2.120 Yates analysis 2^{4-1}

Trials	Response	(1)	(2)	(3)	Average effects (3)/4	Estimated effects
(1)	4.2	7.2	15.1	28.8	–	T
ad	3.0	7.9	13.7	-6.8	-1.70	A
bd	5.0	6.8	-3.3	0.8	0.20	B
ab	2.9	6.9	-3.5	-2.0	-0.50	AB+CD
cd	4.0	-1.2	0.7	-1.4	0.35	C
ac	2.8	-2.1	0.1	-0.2	-0.05	AC+BD
bc	4.6	-1.2	-0.9	-0.6	-0.15	BC+AD
abcd	2.3	-2.3	-1.1	-0.2	-0.05	D
Total	28.8	–	–	–	–	–

The *Yates* procedure as demonstrated in Example 2.31 was used to estimate the effects. Here, one should do the following: shift k=k′ where k′=k-p. Total effects divided by $2^{k'-1}$ offer average effects. Outcomes of *Yates* analysis are shown in Table 2.120.

Example 2.35 [32]

The following chemical reaction has been studied in a chemical laboratory:

A+B+C→D+other products

The reaction takes place with solvent E being present. Five factors, as shown in Table 2.121, were varied in lab conditions. In designing the experiment it has been assumed that interaction effects are not significant. The experiment was therefore done by 2^{5-2} or 1/4-replica each. The design matrix with experimental outcomes is shown in Table 2.122.

Table 2.121 Factors and intervals

Factors	Levels	
	–	+
x_1 **quantity of solvent E, cm^3**	200	250
x_2 **quantity C, mol/molA**	4.0	4.5
x_3 **concentration C, %**	90	93
x_4 **reaction time, h**	1	2
x_5 **quantity B, mol/molA**	3.0	3.5

These generating ratios have been used to construct the matrix: $X_4=X_1X_2X_3$;$X_5=-X_2X_3$

Table 2.122 Fractional factorial experiment 2^{5-2}

No. trials	Design matrix					Operational matrix					Response
	X_1	X_2	X_3	X_4	X_5	x_1	x_2	x_3	x_4	x_5	y_u %
1	–	–	–	–	–	200	4.0	90	1	3	34.4
2	–	–	+	+	+	200	4.0	93	2	3.5	51.6
3	–	+	–	+	+	200	4.5	90	2	3.5	31.2
4	–	+	+	–	–	200	4.5	93	1	3	45.1
5	+	–	–	+	–	250	4.0	90	2	3	54.1
6	+	–	+	–	+	250	4.0	93	1	3.5	62.4
7	+	+	–	–	+	250	4.5	90	1	3.5	50.2
8	+	+	+	+	–	250	4.5	93	2	3	58.6

The following regression coefficients have been obtained by processing the results:

b_0=48.5; b_1=b_1-b_{45}=7.9; b_2=b_2-b_{35}=-2.2; b_3=b_3-b_{25}=6.0;
b_{123}=b_4-b_{15}=0.4; b_{23}=b_5-b_{14}+b_{23}=-0.4; b_{13}=b_{13}+b_{24}=-1.8; b_{12}=b_{12}+b_{34}=0.2.

Example 2.36 [12]

All the thus far formalized steps in defining and performing an experiment are being demonstrated in this example. The process of separating mercury from caustic, as part of the process of extraction in a batch reactor with a mixer, is being tested.

Selection of system response

Mercury content at the process outlet has been accepted as the system response. The response is quantitative and it can be easily measured on an atomic absorption spectrometer with an accuracy of up 1×10^{-6}%.

Selection of factors

These factors affect the mercury content at the process outlet:

1) RPM of the mixer, min^{-1};
2) dwell time of caustic solutions, min;
3) temperature of solution, °C;
4) initial mercury content in caustic, %;
5) quantity of extraction material, g;
6) Solubility of mercury in the solvent, mg/g.

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Previous experiment

A one-factor experiment was done, which showed that, in the assumed experimental region, the quantity of extracted material and its saturation with mercury are of no importance. As in this experiment, a caustic with constant mercury content of 12×10^{-4}% was used, this factor is also excluded from considerations. After this selection the following factors have remained:

x_1 number of mixer rotations, min^{-1}; x_2 temperature of solution, °C;
x_3 dwell time of solution, min.

Description of experimental equipment

Mercury extraction is done in the lab glass reactor consisting of a thick glass vessel 69 mm in diameter and 200 mm high. The vessel is electrically heated with a possibility of regulation of ±1 °C. Mixing is done by a glass mixer with two blades and a possibility of regulating rotation from 250 to 4000 min^{-1}. The reactor construction makes both the batch and the continual operations possible. Industrial nonfiltered caustic solution of 43 to 46% is used in the experiment. Mercury content in such a solution has been from 8×10^{-4} to 18×10^{-4}%.

Definition of the problem

Find extraction conditions of mercury from caustic solution so that at the process outlet we get the minimal mercury content.

Factor space
These limits of factors were selected for all factors based on previous experiments and theoretical knowledge:

$1000 \le x_1 \le 4000\ \text{min}^{-1}$ $\quad 70 \le x_2 \le 130\ °\text{C}$ $\quad 0 \le x_3 \le 100\ \text{min}$

Selection of null level
This experimental center was suggested based on previous information:

$x_{10}=2500\ \text{min}^{-1}$ $\quad x_{20}=100\ °\text{C}$ $\quad x_{30}=45\ \text{min}$

Selection of variation interval
These variation intervals were chosen to realize the basic experiment:

$\Delta x_1=500\ \text{min}^{-1}$ $\quad \Delta x_2=10\ °\text{C}$ $\quad \Delta x_3=15\ \text{min}$

Construction of the design matrix
Mathematical modeling of the process has to be done according to the problem definition. *FUFE* is therefore used with double replication of design points. The design matrix with experimental outcomes is shown in Table 2.123

Table 2.123 Full factorial experiment 2^3

Name	x_1	x_2	x_3	Calculated regression coefficients
Basic	2500	100	45	$b_0=2.95\times10^{-4}$; $b_1=0.45\times10^{-4}$;
Variation interval	500	10	15	$b_2=-1.066\times10^{-4}$; $b_3=0.92\times10^{-4}$;
Upper level	3000	110	60	$\widehat{y} = 2,95\cdot10^{-4} + 0,45\cdot10^{-4}X_1 - 1,066\cdot10^{-5}X_2 + 0,92\cdot10^{-4}X_3$
Lower level	2000	90	30	

Trials	X_0	Design matrix			Operational matrix			Response × 10^4		
		X_1	X_2	X_3	x_1	x_2	x_3	y_{u1}	y_{u2}	$\bar{y}_u$
1	+	+	+	+	3000	110	60	1.09	0.71	0.90
2	+	−	+	+	2000	110	60	1.34	0.94	1.14
3	+	+	−	+	3000	90	60	3.07	2.65	2.86
4	+	−	−	+	2000	90	60	3.42	3.02	3.22
5	+	+	+	−	3000	110	30	2.90	2.50	2.70
6	+	−	+	−	2000	110	30	3.01	2.59	2.80
7	+	+	−	−	3000	90	30	3.74	3.34	3.54
8	+	−	−	−	2000	90	30	6.64	6.26	6.45

A check of statistical significance must be done for the calculated regression coefficients and a check of lack of fit for the regression model. Both checks are a subject of statistical analysis that will be elaborated in more detail in the next chapter. The check of the obtained regression model has shown that it is inadequate, so that we have to reduce variation intervals of factors and increase the number of design-point replications.

Example 2.37 [12]

The effects of temperature, solution concentration and duration of the process on the crystallization speed of aluminum fluorid (AlF_3) have been tested in industrial conditions of its production. Average crystallization of AlF_3 in %/h has been taken as the system response. *FUFE* 2^3 with three parallel design points in the experimental center has been used as a design of the experiment. These three parallel design points are used to estimate experimental error that is necessary for checking the significance of regression coefficients and lack of fit of the obtained regression. All design points have not been replicated in this case as the experiment has been done in industrial conditions. The design matrix with outcomes of the test and data processing are shown Table 2.124.

Table 2.124 Full factorial experiment 2^3

Name		x_1	x_2	x_3	Linear regression
Basic level		90	22	2	
Variation interval		10	4	0.5	$\hat{y} = 9.34 + 0.89X_1 + 2.15X_2 + 1.41X_3$
Upper level		100	26	2.5	
Lower level		80	18	1.5	

Trials	x_0	Design matrix			Operational matrix			Response
		X_1	X_2	X_3	x_1	x_2	x_3	y_u
1	+	+	+	+	100	26	2.5	9.86
2	+	−	+	+	80	26	2.5	9.09
3	+	+	−	+	100	18	2.5	6.35
4	+	−	−	+	80	18	2.5	6.41
5	+	+	+	−	100	26	1.5	15.00
6	+	−	+	−	80	26	1.5	12.02
7	+	+	−	−	100	18	1.5	9.48
8	+	−	−	−	80	18	1.5	6.52
9	+	0	0	0	90	22	2	9.12
10	+	0	0	0	90	22	2	10.30
11	+	0	0	0	90	22	2	5.80

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Example 2.38 [12]

The process of obtaining alkyl sulphonate in an autoclave with a mixer has been studied. Basic reagents were: water solution of sodium hydrosulfite 36-38% and industrial olefin fractions at 240-320 °C. $NaNO_3$ and oxygen from air were used as initiators of the reaction of free radicals. System factors are: x_1 reaction time, h; x_2 temperature of reaction, °C; x_3 mole ratio of sodium hydrosulfite and olefin; x_4 mole ratio of $NaNO_3$ and olefin; x_5 volume ratio of N-propanol and olefin. System response is a yield of alkyl sulfonate as a per cent of theoretical yield. *FRFE* design

2^{5-2} has been used in the experiment with these generating ratios: $X_4=X_1X_2X_3$; $X_5=-X_1X_2$. Determine coefficients of the linear regression model.

Determine aliiased/confounded effects in accord with generating ratios or defining contrasts:

$$1 = X_1X_2X_3X_4 = -X_1X_2X_5 = -X_3X_4X_5$$
$$X_1 = X_2X_3X_4 = -X_2X_5 = -X_1X_3X_4X_5$$
$$X_2 = X_1X_3X_4 = -X_1X_5 = -X_2X_3X_4X_5$$
$$X_3 = X_1X_2X_4 = -X_1X_2X_3X_5 = -X_4X_5$$
$$X_4 = X_1X_2X_3 = -X_1X_2X_4X_5 = -X_3X_5$$
$$X_5 = X_1X_2X_3X_4X_5 = -X_1X_2 = -X_3X_4$$

Taking into account preliminary information on the insignificance of double, triple- and higher-order interactions, we obtain these estimates of main effects:

$b_1=\beta_1+\beta_{234}-\beta_{25}-\beta_{1345}$; $b_2=\beta_2+\beta_{134}-\beta_{15}-\beta_{2345}$;
$b_3=\beta_3+\beta_{124}-\beta_{1235}-\beta_{45}$; $b_4=\beta_4+\beta_{123}-\beta_{1245}-\beta_{35}$; $b_5=\beta_5+\beta_{12345}-\beta_{12}-\beta_{34}$.

The design of experiments with outcomes is shown in Table 2.125.

Table 2.125 Fractional factorial experiment 2^{5-2}

Name		x_1	x_2	x_3	x_4	x_5	Regression coefficients					
Basic level		2.0	100	1.5	0.2	2.0	b_0=27.21			b_1=4.837		
Variation interval		1.0	10	0.5	0.1	1.0	b_2=2.86			b_3=0.81		
Upper level		3.0	110	2.0	0.3	3.0	b_4=0.38			b_5=11.08		
Lower level		1.0	90	1.0	0.1	1.0						
Trials	X_0	Design matrix					Operational matrix					Response
		X_1	X_2	X_3	X_4	X_5	x_1	x_2	x_3	x_4	x_5	y_u
1	+	−	−	−	−	−	1.0	90	1.0	0.1	1.0	14.5
2	+	+	+	−	−	−	3.0	110	1.0	0.1	1.0	18.6
3	+	−	−	+	+	−	1.0	90	2.0	0.3	1.0	13.8
4	+	+	−	+	−	+	3.0	90	2.0	0.1	3.0	51.0
5	+	−	+	+	−	+	1.0	110	2.0	0.1	3.0	23.2
6	+	+	−	−	+	+	3.0	90	1.0	0.3	3.0	41.0
7	+	−	+	−	+	+	1.0	110	1.0	0.3	3.0	38.0
8	+	+	+	+	+	−	3.0	110	2.0	0.3	1.0	17.6

Problem 2.16

Four factors have been varied in testing extraction of hafnium with tributyl phosphate. The experiment has been done by $FUFE\ 2^4$ 1/2-replica design. The design matrix with experimental outcomes is shown in the table 2.126. Determine linear regression coefficients.

Table 2.126 Fractional factorial experiment 2^{4-1}

Trials	X_1	X_2	X_3	X_4	y_u
1	−	−	−	−	0.001675
2	+	−	+	−	0.003465
3	−	−	+	+	0.001540
4	−	+	−	+	0.039750
5	+	+	−	−	0.03800
6	+	−	−	+	0.00470
7	−	+	+	−	0.51000
8	+	+	+	+	0.03975

Problem 2.17

The surface smoothness of the machines in the machine building industry is of the order of a tenth part of a millimeter. Welded parts on such a machine must be processed as the welding may be 2 mm or more thick. To reduce this thickness to the lowest possible measure and to save electrodes and avoid additional machine processing of the welding, an experiment has been designed to model the influence of welding parameters on welding thickness. Factors and variation intervals with design matrix 2^{5-1} are given in Tables 2.127 and 2.128.

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Table 2.127 Factors and variation intervals

Factors	Variation levels			Δx
	−	0	+	
x_1 electrode wear-out rate, m/h	48	56	64	8
x_2 welding rate, m/h	28	34	40	6
x_3 welding step, mm/OB	5	6	7	1
x_4 operational voltage, V	20	21	22	1
x_5 relative position of electrodes	0.250	0.333	0.416	0.083

Table 2.128 Fractional factorial design 2^{5-1}

N_0	X_0	X_1	X_2	X_3	X_4	X_5	X_1X_2	X_1X_3	X_1X_4	X_1X_5	X_2X_3	X_2X_4	X_2X_5	X_3X_4	X_3X_5	X_4X_5	y_a	y_b
1	+	−	−	−	−	+	+	+	+	−	+	+	−	+	−	−	2.20	0.28
2	+	+	−	−	−	−	−	−	−	−	+	+	+	+	+	+	2.97	0.45
3	+	−	+	−	−	−	−	+	+	+	−	−	−	+	+	+	1.60	0.55
4	+	+	+	−	−	+	+	−	−	+	−	−	+	+	−	−	1.98	0.33
5	+	−	−	+	−	−	+	−	+	+	−	+	+	−	−	+	1.90	0.65
6	+	+	−	+	−	+	−	+	−	+	−	+	−	−	+	−	2.20	0.35
7	+	−	+	+	−	+	−	−	+	−	+	−	+	−	+	−	1.04	0.63
8	+	+	+	+	−	−	+	+	−	−	+	−	−	−	−	+	0.82	1.79
9	+	−	−	−	+	−	+	+	−	+	+	−	+	−	+	−	2.31	0.42
10	+	+	−	−	+	+	−	−	+	+	+	−	−	−	−	+	2.73	0.28
11	+	−	+	−	+	+	−	+	−	−	−	+	+	−	−	+	1.90	0.36
12	+	+	+	−	+	−	+	−	+	−	−	+	−	−	+	−	2.38	0.35
13	+	−	−	+	+	+	+	−	−	−	−	−	−	+	+	+	2.03	0.26
14	+	+	−	+	+	−	−	+	+	−	−	−	+	+	−	−	2.27	0.72
15	+	−	+	+	+	−	−	−	−	+	+	+	−	+	−	−	1.17	0.83
16	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	1.55	0.41

Determine regression models for both measured responses.

Problem 2.18

Refinement of waste nitric gases ($NO+NO_2$), is particularly significant from both the point of view of environmental protection and the use of components they contain. A lab plant has been built for this purpose where effects of these factors have been tested:

x_1 consumption of ammonium in the form of liquor ammonia,
x_2 consumption of lye,
x_3 concentration of nitric oxide in gas,
x_4 pH,
x_5 volume of gas flow,
x_6 concentration of salts in circulating lye.

The percentage of absorbed nitric oxides has been measured as a response.

To obtain the mathematical model of the process, 1/4-replica of a full factorial experiment of type 2^6 has been realized. Design points-trials have been done in a completely random order. The Table 2.129 shows conditions and outcomes of doing a 2^{6-2} fractional factorial experiment.

Table 2.129 Fractional factorial design 2^{6-2}

Name	x_1	x_2	x_3	x_4	x_5	x_6	y_u
Basic level	1.25:1	15.4	0.4	8.0	3.0	20	
Variation interval	0.22	1.0	0.1	0.65	1.0	10	
Lower level	1.03:1	14.4	0.3	7.35	2.0	10	
Upper level	1.48:1	16.4	0.5	8.65	4.0	30	
1	–	–	–	–	–	–	45.1
2	+	+	+	+	+	+	50.7
3	–	–	+	+	+	+	42.85
4	+	+	–	–	–	–	43.5
5	+	+	–	+	–	+	33.65
6	–	–	+	–	+	–	45.5
7	+	+	+	–	+	–	56.5
8	–	–	–	+	–	+	37.55
9	–	+	–	–	+	+	35.5
10	+	–	+	+	–	–	58.85
11	–	+	+	+	–	–	50.47
12	+	–	–	–	+	+	40.5
13	+	–	–	+	+	–	56.7
14	–	+	+	–	–	+	38.1
15	+	–	+	–	–	+	59.0
16	–	+	–	+	+	–	58.75

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■ **Problem 2.19**

Describe adequately the process of producing seals by means of a polynomial function. Tensile strength at break kp/cm^2 is the response. Factors of the process are: x_1 contents of parahinom-dioxine in mixture; x_2 contents of solvent (all in grams per 100 grams of butylcaoutchouc). Fractional factorial design 2^{4-1} has been used in the experiment. To assert reproducibility of the experiment, trials have been replicated four times. The design matrix with experimental outcomes is shown in the Table 2.130. Determine the linear and aliiased/confounded effects of even interactions.

Table 2.130 Fractional factorial design 2^{4-1}

Name	x_1	x_2	x_3	x_4
Basic level	1.5	4.25	54	95
Variation interval	1.0	0.5	12	5
Upper level	2.5	4.75	66	100
Lower level	0.5	3.75	42	90

Trials	X_0	Design matrix				Operational matrix				Response				
		X_1	X_2	X_3	X_4	x_1	x_2	x_3	x_4	y_{u1}	y_{u2}	y_{u3}	y_{u4}	$\bar{y}_u$
1	+	−	−	−	−	0.5	3.75	42	90	4.2	3.4	4.0	4.3	3.975
2	+	+	−	+	−	2.5	3.75	66	90	4.7	5.1	5.6	5.3	5.175
3	+	−	−	+	+	0.5	3.75	66	100	4.3	5.2	4.7	5.7	4.975
4	+	−	+	−	+	0.5	4.75	42	100	3.6	3.7	3.9	3.7	3.725
5	+	+	+	−	−	2.5	4.75	42	90	4.5	4.2	4.4	4.6	4.425
6	+	+	−	−	+	2.5	3.75	42	100	4.0	3.6	4.5	4.0	4.050
7	+	−	+	+	−	0.5	4.75	66	90	4.9	4.7	5.1	4.9	4.900
8	+	+	+	+	+	2.5	4.75	66	100	5.0	4.9	5.1	4.9	4.975

Problem 2.20

To control the complex process of cooking by the sulfate process cellulose, by means of a computer, it is necessary to have a mathematical model of the process. To obtain this model for the process of cooking by the sulfate process cellulose from a mixture of soft and hardwood and deciduous trees, we have used a fractional factorial experiment. It included these seven factors: x_1 consumption of active lye, % Na_2O on completely dry wood; x_2 cooking temperature, °C; x_3 hydromodule; x_4 cooking time, min; x_5 percentage of soft and hardwood chips, %; x_6 percentage of normal chips fraction, %; x_7 sulfidity of the lye, %.

The percentage of coarse chips fraction has in all trials been constantly 5%. The contents of normal chips fraction has been changed depending on the design of the experiment. The percentage of fine fraction has been a result of the difference between the coarse and normal fractions and the total chips.

Eleven parameters are analyzed to get the system response: y_1 cellulose yield, calculated for completely dry wood, %; y_2 final contents of lignin in cellulose, %; y_3 degree of delignification of cellulose per permanganate number; y_5 contents of noncooked part in cellulose, %; y_6 pressure decrease by blowdown, kp/cm^2; y_7 resistance to tearing, g; y_8 length of cutting, m; y_9 number of double bonds; y_{10} lagging activity of black lye, g/l; y_{11} density of black lye, g/cm^3.

To perform the experiment, a 1/8 replica of type 2^{7-3} (16 trials) has been chosen with these defining contrasts:

$1=X_1X_2X_3X_4=X_1X_2X_5X_6=X_1X_3X_5X_7=X_1X_4X_6X_7=X_2X_3X_6X_7$
$=X_2X_4X_5X_7=X_3X_4X_5X_6$

All linear effects and the effects of even interactions are being estimated through such a design of experiment:

$X_1X_2=X_3X_4=X_5X_6;$
$X_1X_3=X_2X_4=X_5X_4;$
$X_1X_4=X_2X_3=X_6X_4;$
$X_1X_5=X_2X_6=X_3X_4;$
$X_1X_6=X_2X_3=X_4X_7;$
$X_1X_7=X_3X_5=X_4X_6;$
$X_2X_7=X_4X_5=X_3X_6;$

The associated regression is:

$$\widehat{y} = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 + b_6X_6 + b_7X_7 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{15}X_1X_5 + b_{16}X_1X_6 + b_{17}X_1X_7 + b_{27}X_2X_7 + b_{127}X_1X_2X_7$$

Conditions of varying all factors with the design matrix are shown in Table 2.131. Determine the values of the regression coefficients.

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Table 2.131 Fractional factorial design 2^{7-3}

No.	Design matrix															Responses										
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_1X_2	X_1X_3	X_1X_4	X_1X_5	X_1X_6	X_1X_7	X_2X_7	$X_1X_2X_7$	y_1	y_2	y_3	y_4	y_5	y_6	y_7	y_8	y_9	y_{10}	y_{11}
1	−	−	−	−	−	−	−	+	+	+	+	+	+	+	−	64.5	12.05	63.5	134.1	81.0	7.5	100	8.92	0.61	12.4	1.104
2	+	+	+	+	−	−	−	+	+	+	−	−	−	−	−	42.7	2.54	24.8	73.3	0.0	5.3	107	8.07	1.17	13.4	1.078
3	+	+	−	−	+	+	−	+	−	−	+	+	−	−	−	56.5	2.64	25.2	119.2	8.2	6.1	78	8.50	0.42	22.8	1.152
4	+	−	+	−	+	−	+	−	+	−	+	−	+	−	−	67.8	16.98	80.0	135.3	70.0	6.9	121	9.60	0.40	20.0	1.073
5	+	−	−	+	−	+	+	−	−	+	−	+	+	−	−	59.2	3.50	28.6	116.0	0.0	8.1	124	10.31	0.69	24.3	1.146
6	−	+	+	−	−	+	+	−	−	+	+	−	−	+	−	57.5	2.39	24.0	94.3	34.5	9.5	124	9.93	1.01	3.1	1.058
7	−	+	−	+	+	−	+	−	+	−	−	+	−	+	−	53.3	7.87	47.0	145.0	4.9	10.4	110	9.63	3.40	7.8	1.117
8	−	−	+	+	+	+	−	+	−	−	−	−	+	+	−	75.0	19.61	85.0	96.6	70.0	7.9	110	8.39	0.64	18.6	1.051
9	+	+	−	−	−	−	+	+	−	−	−	−	+	+	+	53.2	2.07	23.0	77.8	2.9	6.6	116	9.90	0.83	26.0	1.151
10	+	−	+	−	−	+	−	−	+	−	−	+	−	+	+	69.6	10.33	56.4	126.5	58.0	6.8	122	9.54	0.82	19.6	1.072
11	+	−	−	+	+	−	−	−	−	+	+	−	−	+	+	51.5	6.58	42.0	144.0	0.0	8.2	105	10.18	1.56	35.6	1.154
12	−	+	+	−	+	−	−	−	−	+	−	+	+	−	+	54.0	11.78	62.4	141.5	15.8	8.0	114	9.01	1.28	9.3	1.058
13	−	+	−	+	−	+	−	−	+	−	+	−	+	−	+	51.1	2.72	25.4	99.5	3.1	7.9	96	11.41	1.17	6.2	1.088
14	−	−	+	+	−	−	+	+	−	−	+	+	−	−	+	61.0	7.04	43.8	116.1	70.0	9.3	104	11.84	1.00	8.3	1.054
15	−	−	−	−	+	+	+	+	+	+	−	−	−	−	+	83.5	18.95	84.0	140.6	80.0	8.3	100	9.55	1.01	18.6	1.109
16	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	45.0	2.75	25.5	80.6	0.0	9.1	117	10.79	1.50	17.3	1.076
17	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	56.0	4.73	33.6	122.0	2.4	8.6	112	11.39	0.73	14.0	1.092
18	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	53.1	5.00	34.8	138.0	2.7	9.8	108	10.57	0.97	14.0	1.096
19	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	56.8	5.14	35.2	92.0	8.5	9.5	103	11.28	1.05	18.6	1.084
20	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	51.1	5.60	37.2	131.6	0.0	8.2	114	10.97	1.85	14.4	1.092

2.3.1.2 Optimality Criterion for Experimental Design

When designing an experiment, a researcher often does not know in advance where in the response surface the optimum is situated and what the shape of that surface is. He therefore tends to choose, first of all, such a design of experiment that which will guarantee him maximum information in the hardest possible situation with a relatively small number of design points. It is natural that in such situations there appears a need to estimate *optimality designs* by using special criteria. It has, first of all, been necessary to determine: what is an optimal design? George E. P. Box and his school are characterized by an empirical intuitive approach of choosing criteria for design optimization. First an *orthogonal* and later a *rotatable* have been suggested as optimal designs. The sense of these criteria could easily be understood intuitively by those researchers who tended to logically think out the experimental methodology. Interesting theoretical results have been obtained from Box's works, especially important for linear designs, and originating from properties of orthogonality and rotatability.

Full factorial experiment and regular fractional replicas as designs, are most efficient for obtaining linear models. Good properties of these designs consist exactly of *orthogonality, rotatability, symmetry* with reference to experimental center and concordance with conditions of *norming*. These properties of designs of experiments may be expressed mathematically thus in this way:

$$\sum_{u=1}^{N} X_{iu} X_{ju} = 0 \text{ for i,j=0,1,...,k;} \tag{2.71}$$

$$\sum_{u=1}^{N} X_{iu} = 0 \quad \text{for i=1,2,...,k;} \tag{2.72}$$

$$\sum_{u=1}^{N} X_{iu}^2 = N \quad \text{for i=1,2,...,k} \tag{2.73}$$

where:
k is number of factors (number of the last design column);
N is number of design points-trials (number of design rows);
u is current number of design points-trials.

Mathematical expression (2.71) corresponds to the condition of orthogonality if the sum of multiplied design signs of any two columns equals zero. Orthogonality of the design facilitates the calculation of independent estimations of regression coefficients. This means dropping all those factors of a design of experiments for which we have regression coefficients and that are statistically insignificant. The expression (2.72) corresponds to the condition of *symmetry* of design with reference to the experimental center. *Norming* of a design of experiment has been defined by relation (2.73). Regression coefficients from designs of experiments that satisfy conditions (2.71)–(2.73), are estimated with a minimal variance (each regression coefficient is estimated based on all N trials with a variance that is N times smaller than the variance or error of the replicated trials).

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A design of experiment is called rotatable if it is insensitive to the rotation of coordinate axes with reference to the experimental center, or if movement of the experimental center in any direction is equivalent. Designs of experiments that satisfy both orthogonality and rotatability do not have minimal variances in determining regression coefficients only, but they are identical too. Orthogonal and rotatable may only be designs of first order; the optimality problem for second-order designs becomes much more complicated. Therefore the first design for second-order models were orthogonal, and later rotatable. In recent years, the concept of D-*optimality,* developed by D. Kiefer, has appeared. He thinks that efficiency of estimates is not determined only by optimal way of processing experimental results but is also conditioned by the optimal distribution of a design point-trials in factor [34]. It has been suggested to include these properties into an optimality design:

- maximal variance value in estimating a response or optimization criterion value;
- volume of elliptical dispersion of parameter estimates.

The designs are called D-optimal if the volume of elliptical dispersion of parameter estimates is minimal. D-optimal designs correspond to designs that minimize the variance of response estimate ($\widehat{y}_u$) in the associated space. In practice, it is difficult to find a design that simultaneously satisfies several optimality criteria. It is therefore recommended in each individual case to:

- choose an optimality criterion;
- choose the most suitable design for the actual case.

A basic requirement in constructing designs of experiments is reduction of the number of experimental trials. Table 2.132 shows the total number of trials (N) for various second-order designs at a different number of factors.

Table 2.132 Number of trials in second-order designs

Number of factors	Number of N design points-trials					Number of regression coefficients
	Orthogonal design	Rotatable design	Hartley design	Kiefer design	Kono design	
2	9	13	7	9	9	6
3	15	20	11	26	21	10
4	25	31	17	72	49	15
5	27*	32*	27*	192	113 (88)	21
6	45	53	29	–	257	28
7	79	92	47	–	577	36

* with halfreplica

It is evident from Table 2.132 that as far as the number of trials is concerned the most economical are *Hartley's* designs. However, attention should also be paid to orthogonal and rotatable ones.

Hartley's design with only seven trials is convenient for k=2. A Konos design may also be good sometimes, and if the number of design points is not limited, we may use Box's rotatable design (N=13). Good results are also achieved by *simplex-summary designs*.

Rotatable design is recommended for k=3. The properties of Hartley's and orthogonal designs are worse, but they may be used when it is necessary to keep a minimal number of design points.

Design B_4, which requires only 24 trials, is recommended for k=4. The design is symmetrical and has certain advantages to the D-optimal design. There is sense in using Hartley's design too.

Hartley's design with only 27 trials should first of all be used for k=5. Box's rotatable design also deserves attention. A comparison of rotatable designs of second order with D-optimal and other designs shows that a rotatable design may be applied where limits of an experimental region are given by a sphere, i.e. in cases when a researcher is only interested in the response surface in the vicinity of the

experimental center. Deficiency of rotatable designs is in experimental design points, not falling into cube crowns but onto the surface of the sphere drawn in the cube. This means that the cube crowns are not used, which greatly influences the accuracy of the obtained model. With an increase in the number of factors, the volumes of unused cube crowns increase, so that for k>5 it is not recommended to use rotatable designs.

Rotatable designs are most efficient for k=3. Rotatable designs of second order are not orthogonal and they do not minimize the variance of estimates of regression coefficients. They are efficient in solving research problems when trying to find an optimum.

Rotatable and orthogonal designs are convenient for application in *composite* designing when designs of second order are constructed after doing the experiment by design of full factorial experiment or fractional replica. Hartley's designs offer a possibility to reduce the number of trials but they are not orthogonal or rotatable. Designs of third order are rarely met in practice as they require a large number of design points.

Summary

The first series of design points-trials of a basic design is preceded by numerous activities meant to select the local domain of factor space. Thereby, limits of factors space that in principle determine the limitations, technoeconomic possibilities and concrete conditions for doing the process, are being estimated. The factor space requires careful analysis of preliminary information on the scope of response change and on curvature of the response surface.

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The local domain of doing an experiment is determined in two stages: determining the basic level and the variation interval. The basic (null) level is a multidimensional point in factor space, given by a combination of factor levels. Construction of a design of experiments is brought down to selection of experimental or design points, symmetrical with reference to a basic level. When defining the basic level it is obligatory to take into account the information on the "best known" point for performing the process, if such information exists.

In the next stage, two variation levels for varying factors in the experiment are determined for each factor. One variation level is called the lower and the other one the upper level. The number that, when added to the basic level gives the upper and when subtracted the lower level, is called the variation interval of the associated factor. To simplify the way of recording conditions of doing an experiment and processing experimental results, axes ratios are such that the value +1 corresponds to the upper, -1 to the lower and zero to the basic level.

Two limitations are imposed on the selection of the factor-variation interval: lower (it may not be smaller than the error of fixing a factor) and upper (upper and lower levels cannot be outside the factor space). In trying to find the optimum in an experimental region, a subregion should be such as to permit the procedure of moving towards the optimum step by step. When determining a model or interpolation, variation intervals include the complete factor space.

When determining the variation interval, we should take into account the accuracy of fixing a factor, curvature of response surface and the range of the response changing. A researcher is greatly helped by twenty seven situations in determining a factor-variation interval. A low accuracy in fixing a factor, by rule, requires a wide variation interval. An average variation interval corresponds to an average accuracy of fixing a factor. A high accuracy of fixing a factor leads to a narrow and average variation interval.

An experiment where all possible combinations of factor or design-point levels are realized is called a full factorial experiment. When the number of variation levels of all factors is two, we have a full factorial experiment of 2^k. Conditions for performing an experiment are presented in tables-design matrices where the rows correspond to different design points-trials and the columns to factors. The design of full factorial 2^2 has a geometric interpretation shaped as a square where crowns correspond to conditions for doing a trial. FUFE 2^3 has the shape of a cube and for $k>3$ we have a hypercube. A type 2^k full factorial experiment has the properties of symmetry, norming, orthogonality and rotatability. Regression coefficients calculated from the outcomes of experiments indicate by their values the degree to which the factors affect the system response. The effect of factors equals double the value of a regression coefficient. We can speak about existence of interaction between two factors in cases when the effect of one factor depends on the level of the other factor. To determine numerical values of interactions, a column of products of associated factors is constructed, which is then manipulated as any other column. The information on the square members may not be drawn out of a full factorial experiment. Columns-vectors for square members coincide both among themselves and with column X_0. The value of free member b_0 includes the effects of square members so that it is basically an aliased/confounded estimate. Estimates of other coefficients are not aliased/confounded. The difference between the number of design points-trials and the number of regression coefficients is great in a full factorial experiment. Therefore a need appeared to construct designs with a smaller number of trials while keeping their optimal properties. Designs where effects of interactions are considered negligible, or designs where they were replaced by new factors, are called fractional replicas. The calculated regression coefficients are, as a rule, estimates of aliiased/confounded effects.

2.3.1.3 **Conclusion after Obtaining Linear Model**

By processing outcomes of a full factorial experiment, we often obtain an adequate linear model that looks like a polynomial of first order. Regression coefficients are the associated partial derivation of a response function by associated factors. As is known, geometrically those are tangents of slope angles of the hypersurface to the corresponding axis. Higher values of regression coefficients by their absolute values correspond to a higher slope angle or to a greater response change at the change of the associated factor. This kind of analysis of a regression model corresponds to the abstract language of mathematics. Its translation into the language of a researcher is called *model interpretation*.

Interpretation of a regression model is complex and it is solved in several stages. The first stage is as follows. A degree of effect of each factor on response is determined. The value of regression coefficient is exactly the quantitative measure of such an effect. The higher this value is, the stronger the effect of the observed factor is. The regression-coefficient sign gives the property of factor effect. A positive sign indicates that with an increase in factor value there is an increase in response value, and a negative sign means a decrease in response value. An interpretation of the regression coefficient signs for research problems of finding the optimum depends on whether we are looking for the maximum or minimum of the response function. When searching for this maximum, an increase of values of all factors having positive regression coefficients is the thing looked for, while an increase in factor values of negative regression coefficients will not contribute to finding the response maximum. When the minimum of a response function is searched for, the situation is completely contrary. One can only say that for the given variation intervals and error reproducibility of an experiment they have no important effect on response.

A change in variation intervals causes changes in regression coefficients. Absolute values of regression coefficients increase with an increase in variation intervals of associated factors. An increase in a factor-variation interval does not change the signs of linear regression coefficients. They may, however, be changed if when moving along the response function gradient they "jump over" the extreme.

In some cases a regression model with real-natural factor dimensions is sought. In that case, we switch from a coded regression model to a real one using transformational expression (2.59). Values of regression coefficients also change with such transformations. Now the possibility of interpreting factor effects on the basis of size and the coefficient sign is lost. Columns or vectors of real factor values in the design of experiment matrix are no longer orthogonal, so that the calculation of regression coefficients is not independent either. A regression model with real factors is acceptable only if the research problem of obtaining the interpolation model with real factors is defined.

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This knowledge offered a basis for a switch to the next stage of interpolation of the regression model. Former conclusions give an idea on the character of factor influence on response. Sources of such conclusions can be: a theory of the studied phenomenon, experiments with similar phenomena or previous design points, etc. If with an increase in temperature we expect an increase in response value, and the regression coefficient has a negative sign, a contradiction is present and it must be solved. Two causes of this contradiction are possible: an error in the experiment or incorrect previous information, i.e. knowledge about the observed phenomenon. One should remember that the experiment is done in the local domain of factor space so that the obtained regression coefficients express factor effects exactly in that part of factor space. Extrapolation of those effects on the rest of factor space is problematic. Theoretical knowledge usually has a more general character so that it may contradict the outcomes from the local domain of factor space. Prior knowledge is often based on single-factor dependencies so that the situation may change when transferring into a multifactor space. This contradiction may be solved by establishing various hypotheses and their experimental checks.

In rare situations when we dispose with sufficient prior theoretical knowledge on the observed phenomenon, one may establish hypotheses on mechanisms of the observed phenomenon, which is the next stage in interpretation of the model. It consists of checking the set hypotheses on the mechanism of the phenomenon and setting up new hypotheses. In this case special attention is paid to the effects of factor interactions and their interpretations.

Assume that in research we have obtained a statistically significant interaction of two factors with a positive sign. This means that simultaneous increase or decrease of both factors brings about a response increase (excluding linear effects). If the double interaction has a negative sign, then any combination of simultaneous increase or decrease of a factor causes an increase in response value. One should remember the rule: if a double interaction has a positive sign, then an increase in response is obtained by simultaneously increasing or decreasing the factor value. To reduce a response it is necessary to change simultaneously both factors in opposite directions. When a double interaction has a negative sign, an increase in response value is obtained by simultaneous changes of both factors in opposite directions. To reduce a response, it is sufficient to increase or decrease both factors.

It is evident that an interpretation of the interaction effect is not so singular as is the case with linear effects. In any case, both variants are at our disposal, and the question is which variant should get an advantage. One should primarily take into account linear effects of the associated factors. If the effect of a double interaction has a plus sign and the associated linear factor effects a minus sign, then negative factor values are chosen ($X_1 = -1$; $X_2 = -1$). A case of different linear effect sign is also possible; then regression coefficient values are compared and the lower value factor is sacrificed. Sometimes in such a situation, the factor whose changes are more difficult to be done in an experiment or which cause higher expense, is sacrificed. An interpretation is more complex in the case of statistical significance of the triple $X_1X_2X_3$. Such an interaction may have a plus sign if the signs of an even number of factors are negative. A triple interaction effect will have a minus sign if an uneven number of factors has the same sign. Such an interpretation may be generalized for interactions of any order. An approach is often used to consider the yield of two factors, conditionally, a single factor, so that a triple interaction is brought down to an even-double one.

It has been mentioned that an interpretation of outcomes means a transfer from one language into another one. Such a transfer facilitates an understanding between statisticians and researchers who jointly work in the study of system optimization. A regression model interpretation is not significant only for an understanding of the mechanism of the process but also for drawing conclusions about solving the problem of optimization.

Conclusions After Obtaining a Mathematical Model

Drawing conclusions after processing experimental outcomes may be very complex depending on the number of factors, on the type of experiment or level of research objective (screening of factors by significance of their effect on response, mathematical modeling and optimization of the process). Complexity of obtained models and

the number of conclusions that may be drawn from them are extremely high and we will therefore limit our analysis to typical cases. It will differ in the lack of fit or nonlack of fit of the model, in significance and insignificance of regression coefficients and in information on position of the optimum.

Adequate linear model
Three cases are possible in this situation:

- all regression coefficients are statistically significant;
- some regression coefficients are statistically significant;
- all regression coefficients are statistically insignificant.

In any of these cases an optimum may be close by, far away or there is no information on its position, i.e. the position of the optimum is not defined.

When the optimum region is close by, three solutions are possible:

- end of research;
- transfer to designs for second-order models;
- movement along the gradient of response function towards optimum.

Transfer to design of experiments for second-order models enables a mathematical description of the optimum and discovery of the extreme. Design of experiments from the second-order models are the subject of the next chapter. Movement along the gradient is applied in the case of a small error in the trial, as it is hard to establish the response yield for a large error. When an optimum is undefined or too far, the solution lies in moving along the gradient.

The second case some regression coefficients are statistically significant. Since movement to an optimum along the gradient is most efficient when all regression coefficients are important, one must choose the solution that makes all regression coefficients statistically significant. One must therefore set up the hypothesis that explains the insignificance of individual coefficients. Insignificance of regression coefficients may be a consequence of wrong choice of variation intervals, inclusion of factors that do not affect the response, (being cautious), large error of trials, etc. The first doubt is removed by an increase of variation intervals for insignificant factors and performance of a new sequence of design points. An increase in variation intervals is usually combined with a shift of the experimental center to the point with the best response value. Insignificant factors are fixed and excluded from the next experiment. The solutions for obtaining significant regression coefficients are:

- increase in number of replicated trials;
- upgrade the design of experiment.

An increase in the number of replicated trials causes a decrease in reproducibility variance or experimental error as well as in the associated variances of regression coefficients. Design points-trials can be replicated in all points of the experiment or in some of them. An upgrade of the design of experiment may be realized by a shift from fractional to full factorial experiment, a switch to bigger replica (from 1/6 to 1/2 replica), a switch to second-order design (when the optimum region is close by), etc.

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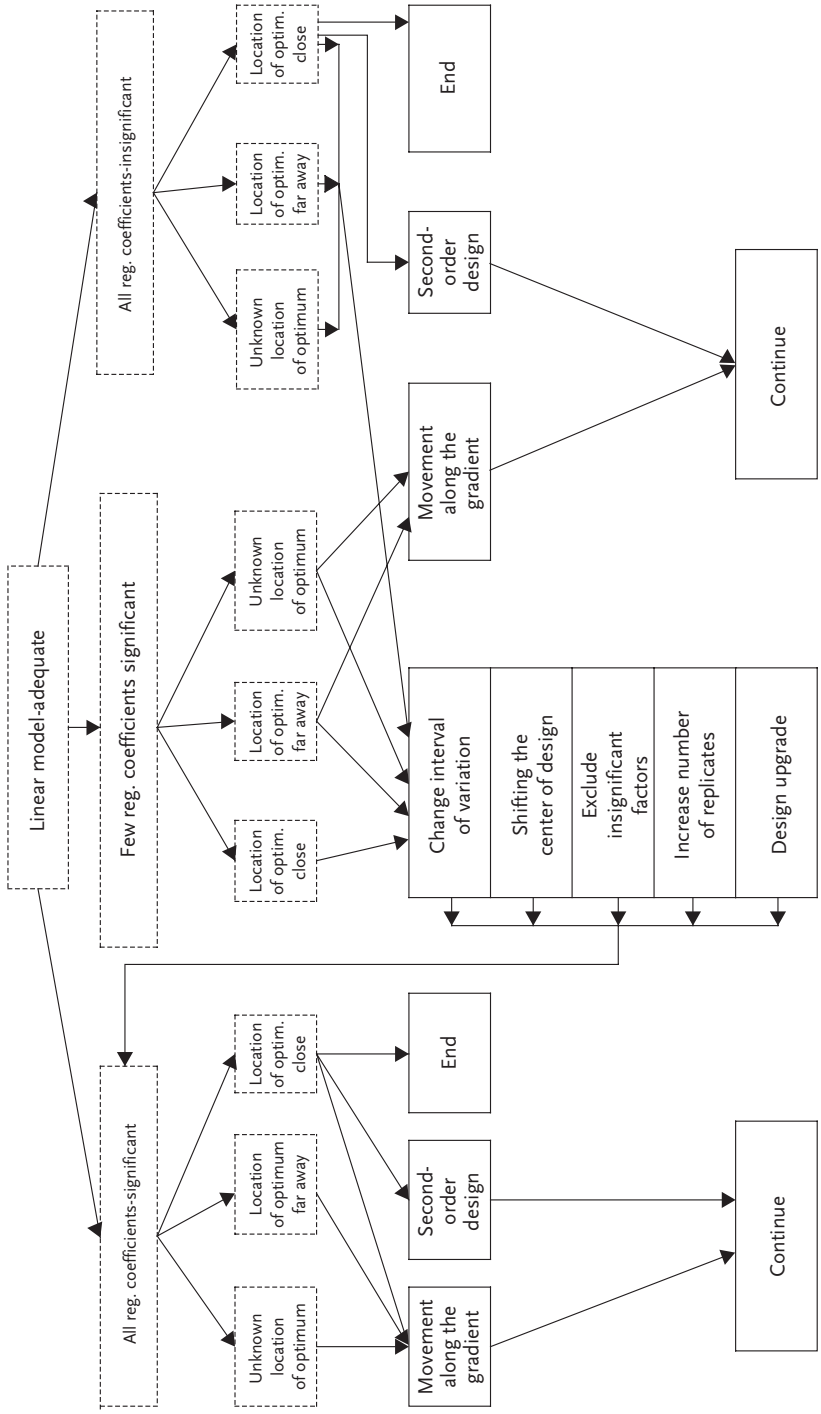

Figure 2.37 Block diagram of search for an optimum for an adequate linear model

Realization of these solutions requires additional experiments, so that it is sometimes unnecessary to follow blindly the rules of movement to optimum by all the factors but to do it by only those factors that are statistically significant.

Finally, if an optimum is close by, one should proceed as in the case when all regression coefficients are statistically significant. Let us analyze the last case: adequate linear model, all regression coefficients statistically insignificant (except b_0). This case mostly occurs as a result of either a larger experimental error or small factor-variation intervals. Possible solutions refer to both an increase in accuracy of the experiment and factor-variation intervals. An increase in accuracy of the experiment may be achieved in two ways: by an improvement in methodology of carrying out and by an increase in the number of replicated trials. The conclusion of this analysis is shown by the block diagram for the case of an adequate linear Fig. 2.37.

Example 2.39 [35]

When determining optimal conditions for the technological process of obtaining man-made fibers from polypropylene, these factors have been chosen: X_1-temperature of melt, °C; X_2-pressure of melt, kp/cm^2; X_3-speed of winding up on roll, m/min; X_4-heating temperature, °C; X_5-speed of drawing-out, m/min and X_6-brevity of drawing-out. Optimization parameter has been the fiber tensile strength. Experimental conditions, design matrix and outcomes of trials are given in Table 2.133. In this example, 1/8 replica of full factorial experiment 2^6 has been used with these generating ratios:

$X_4=X_1X_2X_3$, $X_5=-X_1X_3$ and $X_6=-X_2X_3$

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Table 2.133 Fractional factorial design 2^{6-3}

Name	x_1	x_2	x_3	x_4	x_5	x_6	
Basic level	300	50	2.40	150	0.35	7.2	
Variation level	10	7	0.47	5	0.12	0.3	
Upper level	310	57	2.87	155	0.47	7.5	
Lower level	290	43	1.93	145	0.23	6.9	
Trials	**Design matrix**						**Response**
	X_1	X_2	X_3	X_4	X_5	X_6	y
1	−	−	−	−	−	−	53.4
2	+	−	+	−	−	+	65.3
3	+	−	−	+	+	−	54.2
4	−	+	+	−	+	−	56.2
5	−	+	−	+	−	+	52.8
6	+	+	+	+	−	−	52.2
7	+	+	−	−	+	+	65.1
8	−	−	+	+	+	+	52.8

These regression coefficients were obtained after processing the outcomes:

b_0=56.500 b_3=0.125 b_6=2.500
b_1=2.700 b_4=-3.500 S_b=1.060
b_2=0.0749 b_5=0.575

The obtained linear regression equation is adequate. Three out of six regression coefficients are statistically significant (b_1, b_4 and b_6). There is no information on the position of the optimum. Two variants are feasible in this situation:

- to move along the gradient towards optimum;
- to increase variation intervals of insignificant factors.

Analyze the first variant. The movement to optimum when we have only three out of six significant regression coefficients may be inefficient. Besides, in a 1/8-replica of a full factorial experiment, effects are greatly aliased/confounded up and this mixture is not excluded, so that significant regression coefficients may be estimates of aliasing effects of more significant factors. On the other hand, an increase in factor-variation intervals requires new design points that are very expensive. Moving along the gradient we risk performance of two to three trials only. Therefore, accepting the variant of movement to optimum along the gradient seems reasonable.

The second variant, an increase in variation intervals of insignificant factors with additional design points, is acceptable if movement to optimum appears to be inefficient. A change in variation intervals will require at least eight expensive design points.

Example 2.40 [36]
In a process of separating elements of rare earths on the principle of ionic exchange in solution of amino di carboxylic acid, percentile contents of neodymium in the outlet solution has been used as response. Only two factors have been analyzed during the research: X_1-concentration of inlet solution, % and X_2-pH of inlet solution. The domain of factors was: $X_1 \in 0.5; 3.0$ and $X_2 \in 3; 8$. Previous knowledge indicates that the accuracy of fixing the factor is average, the response surface linear, and the range of response change narrow enough. Therefore, to do the experiment according to block Fig. 2.12 a wide factor variation interval Δx_1=0.5 and Δx_2=1.0, has been chosen, which is 20% of the factor space. Values x_{10}=1.5 and x_{20}=7.0 have been chosen for the basic level. The experiment has been a full factorial one, and the outcomes are shown in Table 2.134.

These regression coefficients are obtained by the processing of results:

b_0=88.0; b_1=-2.0; b_2=-4.5; b_{12}=0.5

The regression equation is adequate and has this form:

$$\widehat{y} = 88.0 - 2.0X_1 - 4.5X_2; \quad S_b = 0.30$$

All regression coefficients are statistically significant and the optimum region is close by, so that the best obtained response value is y=95%. The research objective is to obtain 99 to 100% of neodymium with a minimal number of trials.

Table 2.134 Full factorial experiment 2^2

Number of trials	Yates	Factors				Response
		X_0	X_1	X_2	X_1X_2	y
1	(1)	+	−	−	+	95
2	a	+	+	−	−	90
3	b	+	−	+	−	85
4	ab	+	+	+	+	82

The solution to this problem may be one of the variants:

- movement along the gradient to optimum;
- end of further research;
- upgrading to a design for second-order model.

The first variant of movement along the gradient is most acceptable, as an increase in outlet for several per cent with two to three additional design points is very important for the mentioned procedure of ionic change. This solution is even more acceptable when we know that an upgrading to the existing design towards the second-order model would require at least five trials. The second variant that entails ending of any further research, is unacceptable because the increase in outlets for several per cent is eventually economically advantageous. The third variant is also unacceptable as it requires more trials than the first one.

To finalize the analysis on drawing a conclusion after obtaining an adequate model, it should be pointed out that the research problem of obtaining a model or an interpolation formula has been fulfilled by obtaining an adequate model.

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Inadequate linear model

If a linear model is inadequate it means that the response surface is not approximated to the plane. Apart from Fisher's criterion, which is there to judge the lack of fit of a regression model, inadequacy may also be recognized in this way:

- at least one interaction is statistically significant;
- sum of regression coefficients next to square members $\sum \beta_{ii}$ is statistically significant. The estimate of that sum is given by the difference between b_0 and the response in design center-y_0. When this difference is greater than the experimental error, the hypothesis on statistical insignificance of regression coefficients next to square members cannot be accepted. One should remember that a sum of regression coefficients next to square members may be statistically insignificant although the square effects are significant, because they may have different signs.

Differentiation of significant and insignificant regression coefficients is not important for inadequate models. To obtain an adequate model, we should:

- change the factor variation intervals;
- transfer the design of the experiment center into another point;
- upgrade the design of experiments.

A change in factor-variation interval is the most usual approach, and it requires additional trials. One may sometimes give up obtaining an adequate model, and the possibility of movement along the gradient to optimum is then checked at the expense of additional design points. To move to an optimum along the gradient of an inadequate model, is not a correct approach. Movement along the gradient is preceded by an estimate of curvature of the response surface (based on the sum of regression coefficients next to square members) and a comparison of values of linear regression coefficients with interactions of regression coefficients. When the share of square members and interaction effects is not great, movement to optimum along the gradient is possible.

There is another solution to movement to optimum, i.e. when interaction effects are included in an inadequate regression model and this movement is done by an incomplete second-order model. In that case, the second-order model is analyzed and the gradient direction changed from point to point.

If the optimum region is close by, the research by this model ends and we switch to constructing the design of experiments for the second-order model. Fig. 2.38 shows the block diagram of searching for an optimum for an inadequate linear model.

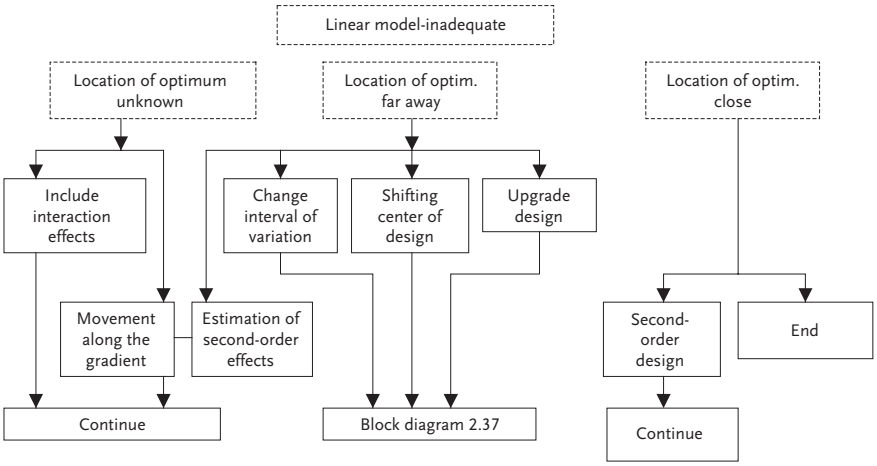

Figure 2.38 Block diagram of search for optimum for an inadequate linear model

Example 2.41 [12]

An optimization of the process of obtaining a pharmaceutical product (carbomethoxysulphonylguanadine) has been done.

The system factors are: X_1-ratio of solvent to basic material, g/l; X_2-temperature of reaction mixture, °C and X_3-reaction time, min. Product yield in per cent is the system response. Experimental conditions and outcomes of trials are given in Table 2.135.

Table 2.135 Full factorial experiment 2^3

Name	x_1	x_2	x_3	Regression coefficients		
Basic level	0.7	135	30	b_0=23.8;	b_1=1.78;	b_2=10.23
Variation interval	0.2	5	15	b_3=9.36;	b_{12}=0.17;	b_{13}=-0.79
Upper level	0.9	140	45	b_{23}=3.77;	b_{123}=1.00;	S_{bi}=0.12
Lower level	0.5	130	15			

Trials	Factors			Response
	X_1	X_2	X_3	
1	+	+	+	46.80
2	+	−	+	20.47
3	−	−	+	16.80
4	−	−	−	5.08
5	+	+	−	24.15
6	+	−	−	8.89
7	−	+	−	16.63
8	−	+	+	46.45

By analyzing the values of regression coefficients, one can state that the following are statistically significant: b_0; b_1; b_2; b_3; b_{13}; b_{23} and b_{123}. A check of lack of fit shows that a linear model is not adequate. In the actual situation, a change in factor-variation interval and a replication of the experiment is acceptable. By a decrease in factor variation X_2 and X_3, the values of the associated regression coefficients b_2 and b_3 will be reduced, as well as coefficients for interactions: b_{13}; b_{23} and b_{123}, to the level when they will become statistically insignificant. There is a reason to transfer the experimental center to the conditions of design points No.1 and No.8. The suggested change in variation interval requires replication of the design of experiment or 8 trials.

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Movement to optimum by an inadequate linear model is also possible in cases when doing the mentioned eight trials is not acceptable. The values of linear regression coefficients are considerably above the values of those for interactions, the more so since linear effects are not aliased/confounded with interaction effects. Although the movement to optimum by an inadequate linear model is mathematically incorrect, it may be accepted in practice with an adequate risk. Note that when trying to optimize a process one should aspire towards both the smallest possible interaction effects and approximate or symmetrical linear coefficients. In problems of interpolation models, the situation is exactly the opposite since it insists on interaction effects, which may be significant.

Interpolation models-inadequate linear model

The first thing one must do when searching for an interpolation model is to include interactions in the model. This is possible when an *unsaturated design* of experiments is used. By introducing interactions there may appear a case where the degrees of freedom are insufficient for a check of lack of fit of the model, and it is therefore necessary to do two to three trials within the experimental region. All

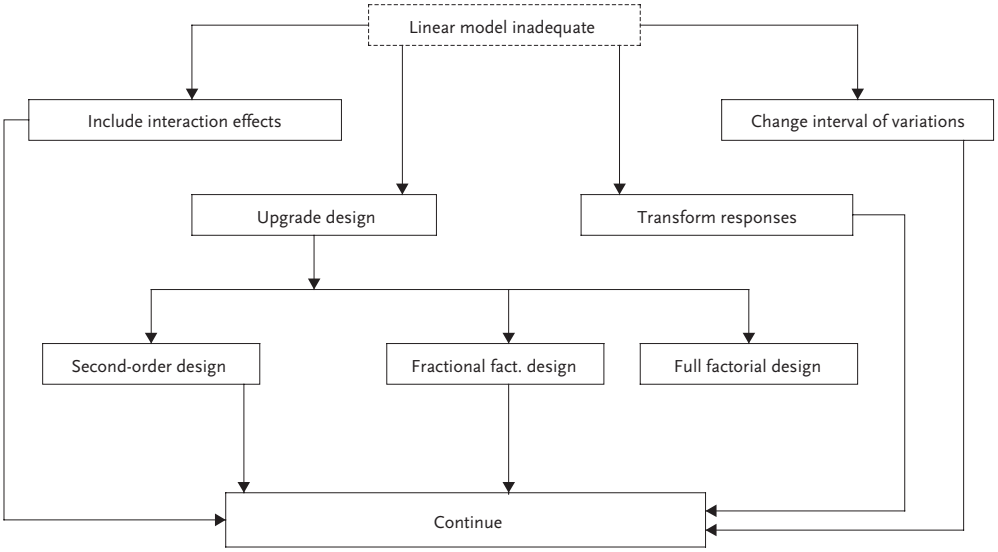

Figure 2.39 Block diagram for obtaining interpolation model, linear model inadequate

other approaches to obtain an interpolation model have to do with realization of new trials, by an upgrading to the basic design of experiment. The same approaches are used as with removal of insignificance of regression coefficients: upgrading to the replica where aliased/confounded effects become clean, upgrading to the full factorial experiment, and to the second-order design. Another unusual approach is factor and response transformation. If by these approaches, an adequate model is not obtained, what remains is to split the experimental region into a number of subregions that will be described by adequate models. This, of course, requires a reduction of factor variation intervals. Fig. 2.39 shows the block diagram for obtaining interpolation model when a linear model is inadequate. When the linear model is adequate, then the research problem is solved.

Example 2.42 [36]
An experiment with these factors was done for mathematical modeling of an extraction process:

x_1-diameter of turbine mixer, mm;
x_2-number of rotations of mixer, min^{-1};
x_3-temperature, °C;
x_4-free acid concentration in water solution, gE/l;
x_5-height of liquid layer, mm and
x_6-ratio of phases in emulsion.

The optimization parameter is the time of complete decomposition in minutes. Experimental conditions, and the matrix of design of experiments with outcomes of

trials are shown in Table 2.136. A 1/4-replica of the full factorial experiment 2^6 was used. The obtained linear model proved to be inadequate. Therefore, three even interactions, which by their absolute values are the biggest and are not interaliased/confounded, have been included in the model.

$$\widehat{y} = 12.16 + 0.53X_1 + 0.53X_2 - 1.38X_3 - 3.22X_4 + 1.44X_5$$
$$-0.62X_6 - 0.84X_1X_4 - 0.50X_1X_6 - 0.78X_2X_4$$

This kind of a regression model is adequate and has been used for designing the industrial extractor.

Table 2.136 Fractional factorial experiment 2^{6-2}

Name	x_1	x_2	x_3	x_4	x_5	x_6	
Basic level	90	600	26	0.40	195	0.8115	
Variation interval	10	100	4	0.29	25	0.0975	
Upper level	100	700	30	0.69	220	0.909	
Lower level	80	500	22	0.11	170	0.714	
Trials	Factors						Response
	X_1	X_2	X_3	X_4	X_5	X_6	Y
1	−	+	+	+	−	−	7.00
2	−	−	−	−	+	−	16.50
3	−	−	−	+	−	−	9.50
4	−	−	+	+	+	+	9.00
5	+	+	+	+	+	+	7.75
6	+	−	−	+	+	+	10.75
7	−	+	−	+	+	+	11.50
8	+	−	−	−	−	+	13.25
9	+	+	−	+	−	−	8.50
10	−	+	+	−	+	−	14.00
11	−	−	+	−	−	+	9.25
12	+	−	+	−	+	−	17.25
13	+	+	+	−	−	+	14.50
14	+	+	−	−	+	−	22.0
15	−	+	−	−	−	+	16.25
16	+	−	+	+	−	−	7.50

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Summary

Translation of a model from the abstract mathematical into a researcher's language is called model interpretation. Interpretation is a complex process with several phases. It includes the estimates of the sizes and signs of the linear factor regression coefficients and their interactions, comparison of factor effects, check of previous-preliminary information, and in some cases a check of the hypothesis on the mecha-

nism of the process. An assembly of actions and experimental situations is reduced to typical cases, which differ by lack of fit and inadequacy of the model, by significance and insignificance of regression coefficients, by position of the optimum, etc.

For an adequate linear model with statistically significant regression coefficients, these decisions are possible: movement to optimum, second-order design, end of research. If part of a regression coefficients is insignificant, then there exist several activities for transforming them into statistically significant ones: change of variation interval, shift of design center, rejection of insignificant factors, increasing number of parallel trials, upgrading of design. Besides, a possibility exists to move to the optimum along the gradient, and if the optimum region is close by, then an upgrading to design towards a second-order design or termination of research are the other options.

Finally, when all regression coefficients are insignificant: second-order design or termination of research (optimum region close by) is realized, procedures are applied for obtaining significant regression coefficients (optimum region far off or its position is undefined). Research is terminated for an inadequate linear model if the optimum region is close by or a second-order design is realized. Change of variation interval, shift of design center, upgrading design, movement along the gradient are all activities for any other position of the optimum. Another possibility is to include interaction effects in the model and then move towards the optimum by means of an incomplete second-order model.

If the experimental objective is to obtain an interpolation model, an adequate linear model is the solution. In the case of an inadequate linear model, one of the following activities is undertaken: inclusion of interaction effects into the model, upgrading the design, transformation of variables, change of variation intervals.

2.3.2
Second-order Rotatable Design (Box-Wilson Design)

Second-order designs are used in practice in situations when the linear model is insufficient for a mathematical description of a research subject with an adequate precision. Then a mathematical model in the form of a second-order polynomial is formed. When describing a response surface by a second-order equation, varying a factor on only two levels does not offer the necessary information. Hence, an experiment is designed so that factors are varied on three or more levels. One of such designs is the *second-order rotatable design* (Box-Wilson design). These designs are particularly interesting for k=3 and k=5 in conditions of *composite* designing (upgrading of designs of lower order).

With second-order rotatable designs, we upgrade a *FUFE* design or its fractional replica (usually half-replica) to get a second-order design by adding a certain number of " *starlike/axial/star"* and " *null/centerpoints*"points to the " *core*". Use of a *FUFE* matrix as "*core*" of a second-order rotatable design for k<5, and half-replica for k>5 is recommended. Starlike points are located on coordinate axes at distance from the experimental center, with taking into account of the rotatability condition (for *FUFE* as "*core*") [30].

$$\alpha=2^{k/4} \tag{2.74}$$

If a fractional replica of type 2^{k-p} has been chosen as the "*core*" of rotatable design, another expression is used :

$$\alpha=2^{(k-p)/4} \tag{2.75}$$

The rotatability condition is defined by these relations:

$$\sum_{1}^{N} X_{iu}^2 = N \times \lambda_2 \text{ for i=1.2,...,k} \tag{2.76}$$

$$\sum_{1}^{N} X_{iu}^4 = 3\sum_{1}^{N} X_{iu}^2 X_{ju}^2 = 3N\lambda_4 \text{ for i, j=1.2,...,k} \tag{2.77}$$

where λ_2 and λ_4 are constants. For k=2 or k=4 these constants are linked by ratio:

$$\lambda_4^* = \frac{\lambda_4}{\lambda_2^2} = \frac{kC}{k+2} \tag{2.78}$$

where C is determined by formula (2.84).

Conditions (2.76) and (2.77) define independence of the design from rotation of coordinates. When selecting the "*null/centerpoints* " points (points in experimental center) take into consideration a check of lack of fit of the model, an estimate of experimental error and conditions of uniformity [37]. Centerpoints are created by setting all factors at their midpoints. In coded form, centerpoints fall at the all-zero level. The centerpoints act as a barometer of the variability in the system. All the necessary data for constructing the rotatable design matrix for k≤7 are in Table 2.137. This kind of designing is called *central*, because all experimental points are symmetrical with reference to the experimental center. This is shown graphically for k=2 and k=3 in Fig. 2.40.

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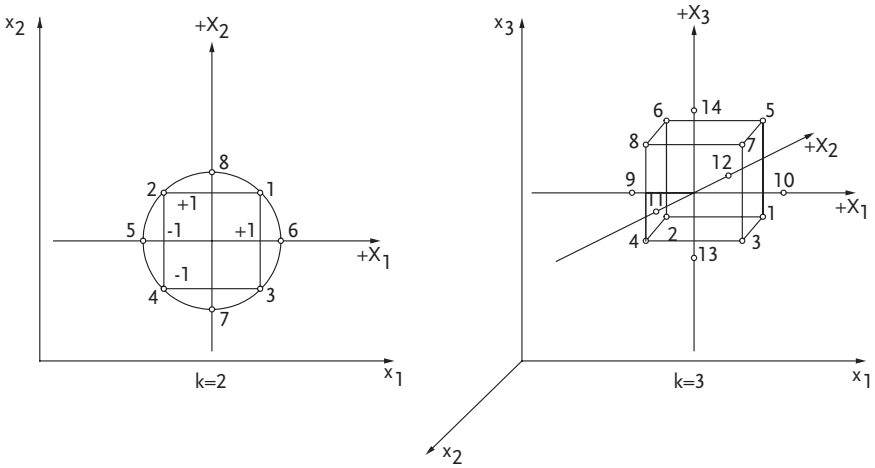

Figure 2.40 Distribution of rotatable design points

Table 2.137 Construction of rotatable design

Number of factors- k	Number of "core" points n_j	Number of "starlike" points n_α	Number of "null" points n_0	Coded values α	Total number of trials- N	Notes on design "core"
2	4	4	5	1.414	13	–
3	8	6	6	1.682	20	–
4	16	8	7	2.000	31	–
5	32	10	10	2.378	52	–
5	16	10	6	2.000	32	Half-replica
6	64	12	15	2.828	91	–
6	32	12	9	2.378	53	Half-replica
7	128	14	21	3.333	163	–
7	64	14	14	2.828	92	Half-replica

Total number of design points N of a rotatable design is determined from:

$$N = 2^k + 2k + n_0 = n_j + n_\alpha + n_0 \tag{2.79}$$

Design matrices of *central composite rotatable designs (CCRD)* for k=2, k=3 and k=5 are shown in Tables 2.138 – 2.140. By using relation (2.59), which connects coded and real factor values, we switch from design matrix to operational matrix, Table 2.138.

Table 2.138 Central composite rotatable design $2^2 + 2x2 + 5$

Number of trials	Design matrix		Operational matrix		Response	Predicted values	
	X_1	X_2	x_1 %	x_2 %	y_u	$\widehat{y}_u$	$\left(y_u - \widehat{y}_u\right)^2$
1	+	+	2.4	100	50	49.9	0.01
2	–	+	1.4	100	67	65.2	3.24
3	+	–	2.4	40	60	62.4	5.76
4	–	–	1.4	40	70	70.6	0.36
5	-1.414	0	1.2	70	70	68.1	3.61
6	+1.414	0	2.6	70	50	51.5	2.25
7	0	-1.414	1.9	28	72	70.6	1.96
8	0	+1.414	1.9	112	56	57.5	2.25
9	0	0	1.9	70	62	64.0	4.00
10	0	0	1.9	70	64	64.0	0.00
11	0	0	1.9	70	68	64.0	16.00
12	0	0	1.9	70	64	64.0	0.00
13	0	0	1.9	70	62	64.0	4.00
					$\sum_1^{13} y_u = 815$		$\sum_1^{13} \left(y_u - \widehat{y}_u\right)^2 = 43,44$

Table 2.139 Central composite rotatable design $2^3 + 2x3 + 6$

Number of trials	X_1	X_2	X_3	$\bar{y}_u$
1	+	+	+	20.67
2	+	+	−	17.32
3	+	−	+	16.90
4	+	−	−	16.72
5	−	+	+	15.54
6	−	+	−	15.39
7	−	−	+	15.22
8	−	−	−	15.13
9	-1.68	0	0	15.19
10	1.68	0	0	17.01
11	0	-1.68	0	13.96
12	0	1.68	0	15.76
13	0	0	-1.68	15.48
14	0	0	1.68	15.96
15	0	0	0	15.97
16	0	0	0	16.00
17	0	0	0	15.10
18	0	0	0	14.90
19	0	0	0	14.78
20	0	0	0	16.07

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Regression coefficients are calculated after constructing the operational matrix by these equations:

$$b_0 = \frac{2A}{N\left[(\lambda_4^*)^2(k+2)\sum_1^N y_u - C\times\lambda_4^*\sum_1^N\sum_1^k X_{iu}^2\times y_u\right]} \tag{2.80}$$

$$b_i = \frac{\sum_1^N X_{iu}\times y_u}{N-n_0} \tag{2.81}$$

$$b_{ij} = \frac{C^2}{N\lambda_4^*}\sum_1^{n_j} X_{iu}X_{ju}y_u \tag{2.82}$$

$$b_{ii} = \frac{AC^2}{N}\left[(k+2)\lambda_4^* - k\right]\sum_1^N X_{iu}^2 y_u + \frac{AC^2}{N}\left(1-\lambda_4^*\right)\sum_1^N\sum_1^N X_{iu}^2 y_u - \frac{2AC}{N}\lambda_4^*\sum_1^N y_u \tag{2.83}$$

where:

$$C = \frac{N}{N-n_0}; A = \frac{1}{2\lambda_4^*[(k+2)\lambda_4^* - k]} \tag{2.84}$$

Table 2.140 Central composite rotatable design $2^{5-1} + 2x5 + 6$

No. of trials	X_1	X_2	X_3	X_4	X_5	y_u
1	+	+	+	+	+	31.1
2	−	+	+	+	−	30.8
3	+	−	+	+	−	27.1
4	−	−	+	+	+	29.1
5	+	+	−	+	−	21.6
6	−	+	−	+	+	20.8
7	+	−	−	+	+	24.8
8	−	−	−	+	−	17.8
9	+	+	+	−	−	27.2
10	−	+	+	−	+	29.4
11	+	−	+	−	+	31.1
12	−	−	+	−	−	30.8
13	+	+	−	−	+	23.4
14	−	+	−	−	−	22.2
15	+	−	−	−	−	23.4
16	−	−	−	−	+	22.3
17	-2	0	0	0	0	23.0
18	2	0	0	0	0	31.7
19	0	-2	0	0	0	28.3
20	0	2	0	0	0	26.8
21	0	0	-2	0	0	18.8
22	0	0	2	0	0	35.8
23	0	0	0	-2	0	25.2
24	0	0	0	2	0	27.0
25	0	0	0	0	-2	23.2
26	0	0	0	0	2	28.5
27	0	0	0	0	0	28.3
28	0	0	0	0	0	32.1
29	0	0	0	0	0	28.0
30	0	0	0	0	0	29.4
31	0	0	0	0	0	34.5
32	0	0	0	0	0	35.1

When estimating the significance of regression coefficients, these equations are used:

$$S^2_{b_0} = \frac{2A\lambda^*_4(k+2)}{N} S^2_{\bar{y}} \tag{2.85}$$

$$S^2_{b_i} = \frac{S^2_{\bar{y}}}{N - n_0} \tag{2.86}$$

$$S^2_{b_{ij}} = \frac{C^2}{N} S^2_{\bar{y}} \tag{2.87}$$

$$S^2_{b_{ii}} = \frac{AC^2[(k+1)\lambda^*_4 - (k-1)]}{N} S^2_{\bar{y}} \tag{2.88}$$

The hypothesis on lack of fit of second-order model is checked by means of Eqs. (2.172); (2.173); (2.133) and (2.135).

Calculation of regression coefficients may be transformed into a simpler form:

$$b_0 = a_1 \sum_1^N y_u - a_2 \sum_1^k \sum_1^N X^2_{iu} \times y_u \tag{2.89}$$

$$b_i = a_3 \sum_1^N X_{iu} \times y_u \tag{2.90}$$

$$b_{ij} = a_4 \sum_1^{n_j} X_{iu} X_{ju} y_u \tag{2.91}$$

$$b_{ii} = a_5 \sum_1^N X^2_{iu} \times y_u + a_6 \sum_1^k \sum_1^N X^2_{iu} \times y_u - a_7 \sum_1^N y_u \tag{2.92}$$

where: a_1, ..., a_7 are coefficients as determined from Table 2.141.

Table 2.141 Coefficients values $a_1 \div a_7$

Number of factors	Number of trials	Coefficients						
k	N	a_1	a_2	a_3	a_4	a_5	a_6	a_7
2	**13**	0.2000	0.1000	0.1250	0.2500	0.1250	0.0187	0.1000
3	**20**	0.1663	0.0568	0.0732	0.1250	0.0625	0.0069	0.0568
4	**31**	0.1428	0.0357	0.0417	0.0625	0.0312	0.0037	0.0357
5*	**32**	0.1591	0.0341	0.0417	0.0625	0.0312	0.0028	0.0341
5	**52**	0.0988	0.0191	0.0231	0.0312	0.0156	0.0015	0.0191
6*	**53**	0.1108	0.0187	0.0231	0.0312	0.0156	0.0012	0.0187
6	**91**	0.0625	0.0098	0.0125	0.0156	0.0078	0.0005	0.0098
7*	**92**	0.0730	0.0098	0.0125	0.0156	0.0078	0.0005	0.0098
7	**163**	0.0398	0.0052	0.0066	0.0078	0.0039	0.0002	0.0052

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* With half-replica

Equations. (2.89) to (2.92) have these forms for k=3:

$$b_0 = 0.1663 \sum_1^{20} y_u - 0.0568 \sum_1^3 \sum_1^{20} X^2_{iu} y_u$$

$$b_i = 0.0732 \sum_1^{20} X_{iu} y_u$$

$$b_{ij} = 0.1250 \sum_{1}^{8} X_{iu} X_{ju} y_u$$

$$b_{ii} = 0.0625 \sum_{1}^{20} X_{iu}^2 y_u + 0.0069 \sum_{1}^{3} \sum_{1}^{20} X_{iu}^2 y_u - 0.0568 \sum_{1}^{20} y_u$$

Example 2.43 [30]

The outcome of research in the field of textile tissues is shown in Table 2.138. The research included two factors: X_1-reagent concentration, %; X_2-temperature, °C. Variation intervals are shown in Table 2.142.

Table 2.142 Factor variation intervals

Factors	Variation levels					Variation intervals Δx
	-1.414	-1	0	+1	+1.414	
x						
x_1, %	1.2	1.4	1.9	2.4	2.6	0.5
x_2, °C	28	40	70	100	112	30

Factor values in "*starlike*" points have been determined by relation (2.59):

$$X_1 = \frac{x_1 - 1.9}{0.5}; \qquad X_2 = \frac{x_2 - 70}{30}; \quad -1.414 = \frac{x_1 - 1.9}{0.5} \Rightarrow x_1 = 1.2;$$

$$-1.414 = \frac{x_2 - 70}{30} \Rightarrow x_2 = 28$$

By processing outcomes, we obtain estimates of regression coefficients for the second-order regression model:

$$\widehat{y}_u = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2 + b_{11} X_1^2 + b_{22} X_2^2 \tag{2.93}$$

Regression coefficients are calculated by relations (2.89) and (2.92) and by using Table 2.141.

$$b_0 = 0.2 \sum_{1}^{13} y_u - 0.1 \sum_{1}^{2} \sum_{1}^{6} X_{iu}^2 y_u = 0.2 \times 815 - 0.1(487 + 503) = +64.0$$

$$b_1 = 0.125 \sum_{1}^{6} X_{1u} y_u = 0.125(50 - 67 + 60 - 70 - 1.414 \times 70 + 1.414 \times 50)$$

$$= -5.875$$

$$b_2 = \sum_{1}^{6} X_{2u} y_u = 0.125(50 + 67 - 70 - 1.414 \times 72 - 60 + 1.414 \times 56) = -4.500$$

$$b_{12} = 0.25 \sum_{1}^{4} X_{1u} X_{2u} y_u = 0.25(50 - 67 - 60 + 70) = -1.750$$

$$b_{11} = 0.125 \sum_{1}^{6} X_{1u}^2 y_u + 0.0187 \sum_{1}^{2} \sum_{1}^{6} X_{iu}^2 y_u - 0.100 \sum_{1}^{13} y_u$$

$$= 0.125(50 + 67 + 60 + 70 + 2 \times 70 + 2 \times 50) + 0.0187(487 + 503) - 0.100 \times 815 = -2.112$$

$$b_{22} = 0.125\sum_{1}^{6} X_{2u}^2 y_u + 0.0187\sum_{1}^{2}\sum_{1}^{6} X_{iu}^2 y_u - 0.100\sum_{1}^{13} y_u$$

$$= 0.125(50 + 67 + 60 + 70 + 2 \times 72 + 2 \times 56) + 0.0187 \times 990 - 0.100 \times 815 = 0.112$$

Hence Eq. (2.93) becomes:

$$\widehat{y} = 64.00 - 5.88X_1 - 4.50X_2 - 1.75X_1X_2 - 2.11X_1^2 + 0.11X_2^2 \qquad (2.94)$$

Lack of fit of the obtained regression model, for the case of rotatable designing with trials replicated only in design center, is checked by the relation (2.173):

$$S_{AD}^2 = \frac{S_R - S_E}{f_{AD}} = \frac{\sum_{1}^{13}\left(y_u - \widehat{y}_u\right)^2 - \sum_{1}^{5}\left(y_{0j} - \bar{y}_0\right)^2}{13-6-4} = \frac{43.44-24.00}{3} = 6.28$$

$$S_{\bar{y}}^2 = \frac{\sum_{1}^{5}\left(y_{0j} - \bar{y}_0\right)^2}{n_0 - 1} = 6.00 \quad ; \quad F_R = \frac{S_{AD}^2}{S_{\bar{y}}^2} = 1.05$$

If degrees of freedom are f_{AD}=13-6-4=3; f_E=n_0-1=5-1=4 and 1-α=95 %, F_T=6.59 is obtained from Table E. Since F_T>F_R, we may consider the regression equation (2.94) adequate. The significance of regression coefficients is checked by the expressions (2.85) to (2.88).

$$S_{b_0}^2 = 0.20 \times S_{\bar{y}}^2 = 0.20 \times 6.0 = 1.20 \Rightarrow S_{b_0} = 1.10$$

$$S_{b_i}^2 = 0.125 \times S_{\bar{y}}^2 = 0.125 \times 6.0 = 0.75 \Rightarrow S_{b_i} = 0.87$$

$$S_{b_{ii}}^2 = 0.1438 \times S_{\bar{y}}^2 = 0.1438 \times 6.0 = 0.86 \Rightarrow S_{b_{ii}} = 0.93$$

$$S_{b_{ij}}^2 = 0.25 \times S_{\bar{y}}^2 = 0.25 \times 6.0 = 1.50 \Rightarrow S_{b_{ij}} = 1.23$$

$$\Delta b_0 = \pm 2 \times S_{b_0} = \pm 2.20; \quad \Delta b_i = \pm 2 \times S_{b_i} = \pm 1.74;$$

$$\Delta b_{ii} = \pm 2 \times S_{b_{ii}} = \pm 1.86; \quad \Delta b_{ij} = \pm 2 \times S_{b_{ij}} = \pm 2.46.$$

By comparing absolute values of regression coefficients with calculated intervals, we may assert with 95% confidence level that all regression coefficients except b_{12} and b_{22} are statistically significant. The regression equation becomes:

$$\widehat{y} = 64.00 - 5.88X_1 - 4.50X_2 - 2.11X_1^2 \qquad (2.95)$$

Example 2.44

This example shows a second-order rotatable design for three factors (k=3). The design matrix, in accord with Table 2.139 and Table 2.137 contains 20 design points in total, eight design points in the design core (n_f=8) and six replicate design points in the design center (n_0=6). Intervals and levels of variation of the three factors are shown in Table 2.143.

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Table 2.143 Factor variation intervals

Factors	Variation levels					Variation intervals
	-1.682	-1	0	+1	+1.682	Δx
x_1-temperature, °C	130	140	155	170	180	15
x_2-pressure, kp/cm^2	3.2	10	20	30	36.8	10
x_3-time, h	10	30	60	90	110	30

The objective of the research has been the optimization of adhesion process of thermoplastic polymer on pressing at a higher temperature. Tensile strength (y_u) has been analyzed as the optimization criterion. The linear model obtained after the first eight design points was inadequate. *FUFE* design has therefore been upgraded to a second-order rotatable design. Regression coefficients were determined from experimental data for the next regression equation:

$$\widehat{y}_u = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2$$

Regression coefficients are determined from relations (2.89) to (2.92):

b_0=0.1663 × 319.01-0.0568(221.02+217.00+221.86)=+15.47;

$$b_1 = 0.0732\sum_1^{10} X_{1u}y_u = 0.0732(20.67 + 17.32 + 16.90 + 16.72 - 15.54 - 15.39 - 15.22 - 15.13 - 1.683 \times 15.19 + 1.683 \times 17.01) = +0.980;$$

$$b_2 = 0.0732\sum_1^{10} X_{2u}y_u = +0.584$$

$$b_3 = 0.0732\sum_1^{10} X_{3u}y_u = +0.326$$

$$b_{12} = 0.125\sum_1^{8} X_{1u}X_{2u}y_u = 0.125(20.67 + 17.32 - 16.90 - 16.72 - 15.54 - 15.39 + 15.22 + 15.13) = +0.474;$$

$$b_{13} = 0.125\sum_1^{8} X_{1u}X_{3u}y_u = +0.411;$$

$$b_{23} = 0.125\sum_1^{8} X_{2u}X_{3u}y_u = +0.404;$$

$$b_{11} = 0.0625\sum_1^{10} X_{1u}^2y_u + 0.0069\sum_1^{3}\sum_1^{10} X_{1u}^2y_u - 0.0568\sum_1^{20} y_u = 0.0625(132.89 + 2.83 \times 15.19 + 2.83 \times 17.01) + 0.0069(221.01 + 217.00 + 221.86) - 0.0568 \times 319.01 = +0.248;$$

$$b_{22} = 0.0625 \sum_{1}^{10} X_{2u}^2 y_u - 13.566 = 0.0625 \times 217.00 - 13.566 = -0.003;$$

$$b_{33} = 0.0625 \sum_{1}^{10} X_{3u}^2 y_u - 13.566 = 0.0625 \times 221.86 - 13.566 = +0.300$$

Regression equation is as follows:

$$\begin{aligned}\widehat{y}_u = {} & 15.470 + 0.980X_1 + 0.584X_2 + 0.326X_3 + 0.474X_1X_2 + 0.411X_1X_3 \\ & + 0.404X_2X_3 + 0.248X_1^2 - 0.003X_2^2 + 0.3X_3^2\end{aligned} \tag{2.96}$$

The statistical significance of regression coefficients in Eq. (2.96) is checked by reproducibility variance ($S_{\bar{y}}^2 = 0.32$). By using expressions (2.155) to (2.158) we get:

$$\begin{aligned}&\Delta b_0 = \pm 0.816; \quad && S_{\bar{y}} = \pm 0.462; \\ &\Delta b_i = \pm 0.542; && S_{\bar{y}} = \pm 0.307; \\ &\Delta b_{ij} = \pm 0.708; && S_{\bar{y}} = \pm 0.401; \\ &\Delta b_{ii} = \pm 0.526; && S_{\bar{y}} = \pm 0.298.\end{aligned}$$

By comparing absolute values of regression coefficients with errors in their estimates, it becomes evident that all regression coefficients are statistically significant with 0.95% confidence, except for b_{11} and b_{22}. A check of lack of fit of the obtained regression model proved that it is adequate with 95% confidence ($F_R < F_T$).

Example 2.45

The experiment consisted of 32 design points, six design points replicated in the design center and 16 half-replica design points of type 2^{5-1}. The design of experiments matrix with the outcomes of experiments is shown in Table 2.140. The second-order regression model has this form:

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$$\begin{aligned}\widehat{y}_u = {} & b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 \\ & + b_{14}X_1X_4 + b_{24}X_2X_4 + b_{34}X_3X_4 + b_{15}X_1X_5 + b_{25}X_2X_5 + b_{35}X_3X_5 \\ & + b_{45}X_4X_5 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{44}X_4^2 + b_{55}X_5^2\end{aligned}$$

Regression coefficients are calculated thus:

$$b_0 = 0.1591 \sum_{1}^{32} y_u - 0.0341 \sum_{1}^{5} \sum_{1}^{18} X_{iu}^2 y_u = +31.219;$$

$$b_1 = 0.0417 \sum_{1}^{18} X_{1u} y_u = +0.996;$$

$$b_2 = -0.121; \qquad b_3 = +3.929;$$

$$b_4 = -0.129; \qquad b_5 = +0.904;$$

$$b_{12} = 0.0625 \sum_{1}^{16} X_{1u} X_{2u} y_u = -0.394;$$

$$b_{13} = -0.856; \qquad b_{14} = +0.356; \qquad b_{15} = +0.694;$$
$$b_{23} = +0.044; \qquad b_{24} = +0.001; \qquad b_{25} = -0.331;$$
$$b_{34} = +0.370; \qquad b_{35} = -0.094; \qquad b_{45} = +0.369;$$
$$b_{11} = 0.0312 \sum_{1}^{18} X_{1u}^2 y_u + 0.0028 \sum_{1}^{5} \sum_{1}^{18} X_{iu}^2 y_u - 0.0341 \sum_{1}^{32} y_u = -0.957;$$
$$b_{22} = -0.907; \quad b_{33} = -0.969; \; b_{44} = -1.269; \; b_{55} = -1.332.$$

Thus, this regression equation has been obtained:

$$\begin{aligned} \widehat{y}_u = {} & 31.22 + 1.00X_1 - 0.12X_2 + 3.93X_3 - 0.13X_4 - 0.90X_5 - 0.96X_1^2 \\ & - 0.91X_2^2 - 0.97X_3^2 - 1.27X_4^2 - 1.33X_5^2 - 0.39X_1X_2 - 0.86X_1X_3 \\ & + 0.36X_1X_4 + 0.69X_1X_5 + 0.04X_2X_3 + 0.001X_2X_4 - 0.33X_2X_5 \\ & + 0.37X_3X_4 - 0.09X_3X_5 + 0.37X_4X_5 \end{aligned} \tag{2.97}$$

A check of statistical significance of regression coefficients for reproducibility variance $S_{\bar{y}}^2$=9.76 has given these values:

$$S_{b_0}^2 = 0.1591 \times S_{\bar{y}}^2 = 0.1591 \times 9.76 = 1.553;$$

$$S_{b_i}^2 = 0.0417 \times 9.76 = 0.409; \; S_{b_{ij}}^2 = 0.0625 \times 9.76 = 0.610; \; S_{b_{ii}}^2 = 0.0341 \times 9.76 = 0.323$$

so that:

$$\Delta b_0 = \pm 2S_{b_0} = \pm 2.50; \qquad \Delta b_i = \pm 2S_{b_i} = \pm 1.28;$$
$$\Delta b_{ij} = \pm 2S_{b_{ij}} = \pm 1.56; \qquad \Delta b_{ii} = \pm 2S_{b_{ii}} = \pm 1.14.$$

By comparing absolute values of regression coefficients with their interval estimates, with 95% confidence, these regression coefficients are statistically significant: b_0, b_3, b_{44} and b_{55}. Following this the regression model (2.97) becomes:

$$\widehat{y}_u = 31.22 + 3.93X_3 - 1.27X_4^2 - 1.33X_5^2 \tag{2.98}$$

A check of lack of fit of this model shows that it is adequate with 95% confidence.

Example 2.46 [38]

We should experimentally establish the effect of ingredients in extracted phosphorous acid to the degree of decomposition of flotational concentration of phosphorite (y) and the maximized decomposition rate (y_{max}). Significant factors for decomposition are: x_1-temperature of process C; X_2 – X_5 percentage content of ingredients in phosphorous acid MgO, SO_3, Al_2O_3 and F %, respectively. Basic levels, variation intervals and limits of factor space are given in Table 2.144. Factor space corresponds to the scope of changes in concentration of ingredients in industrially extracted phosphorous acid. Extrapolation outside limits given in Table 2.144 when determining y_{max}, has therefore no sense.

Table 2.144 Factor variation intervals

Factors	Variation levels			Variation intervals Δx
	-2	0	+2	
x_1-temperature, °C	10	50	90	20
x_2-MgO, %	0.3	2.1	3.9	0.9
x_3-SO_3, %	0.0	2.0	4.0	1.0
x_4-Al_2O_3, %	0.59	1.33	2.07	0.37
x_5-F, %	0.25	0.75	1.25	0.25

To obtain the second-order regression model, second-order *CCRD* has been used. The number of design points (trials) for k=5 was 32. The design core has corresponded to half-replica 2^{5-1} with this generating ratio $X_5=X_1X_2X_3X_4$. The value and number of design points in the experimental center $n_0=6$ are determined from Table 2.137. The design of experiments with outcomes is shown in Table 2.145.

Table 2.145 CCRD $2^{5-1}+2\times5+6$

No.	X_1	X_2	X_3	X_4	X_5	y_u	No.	X_1	X_2	X_3	X_4	X_5	y_u
1	+	+	+	+	+	34.7	**17**	-2	0	0	0	0	25.0
2	−	+	+	+	−	40.0	**18**	+2	0	0	0	0	33.3
3	+	−	+	+	−	39.0	**19**	0	-2	0	0	0	49.2
4	−	−	+	+	+	39.2	**20**	0	+2	0	0	0	42.0
5	+	+	−	+	−	26.6	**21**	0	0	-2	0	0	17.5
6	−	+	−	+	+	29.5	**22**	0	0	+2	0	0	41.0
7	+	−	−	+	+	30.0	**23**	0	0	0	-2	0	35.6
8	−	−	−	+	−	34.5	**24**	0	0	0	+2	0	27.2
9	+	+	+	−	−	32.2	**25**	0	0	0	0	-2	39.0
10	−	+	+	−	+	41.4	**26**	0	0	0	0	+2	30.0
11	+	−	+	−	+	33.7	**27**	0	0	0	0	0	35.4
12	−	−	+	−	−	40.9	**28**	0	0	0	0	0	36.4
13	+	+	−	−	+	23.9	**29**	0	0	0	0	0	33.2
14	−	+	−	−	−	33.3	**30**	0	0	0	0	0	32.4
15	+	−	−	−	−	27.7	**31**	0	0	0	0	0	37.7
16	−	−	−	−	+	35.9	**32**	0	0	0	0	0	36.9

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Based on the outcomes in the experimental center, these reproducibility variances have been determined: $S^2_{\bar{y}}=4.466$ with this degree of freedom $f=n_0-1=5$. Regression coefficients have these values:

$b_0=34.41$; $b_1=1.08$; $b_2=-0.15$; $b_3=4.51$; $b_4=-0.54$;
$b_5=-1.30$; $b_{12}=0.15$; $b_{13}=0.26$; $b_{14}=1.61$; $b_{15}=0.05$
$b_{23}=0.74$; $b_{24}=-0.20$; $b_{25}=0.40$; $b_{34}=0.40$; $b_{35}=0.26$;
$b_{45}=0.93$; $b_{11}=-1.5$; $b_{22}=2.66$; $b_{33}=-1.47$; $b_{44}=-0.93$; $b_{55}=-0.15$.

By rejecting statistically insignificant regression coefficients, we get this regression model:

$$\widehat{y}_u = 34.4 + 4.51X_3 - 1.3X_5 - 1.5X_1^2 + 2.66X_2^2 - 1.47X_3^2 + 1.61X_1X_4$$

A check of lack of fit shows that the obtained model is adequate with 95% confidence. This regression model facilitates determining the degree of decomposition of the observed raw material at different temperatures depending on changes in contents of ingredients in the acid. To obtain the maximal degree of decomposition y_{max}, it is necessary to set factors X_2 and X_5 to these values: X_2=+2 and X_5=-2, which is evident from the regression model. The effect of SO_3 (X_3) content in phosphorous acid is defined in the regression model by positive linear and negative square regression coefficients. Optimal contents of this ingredient are determined from the maximal value of y by X_3 and it is 1.533%. The regression model for X_2, X_3 and X_5 has the form:

$$\widehat{y}_u = 52.12 - 1.5X_1^2 + 1.61X_1X_4 \tag{2.99}$$

To determine optimal values of temperature and contents of ingredient Al_2O_3 (X_4), it is necessary to transfer regression (2.99) into canonical form.

Example 2.47 [18]

In Example 2.26, we have obtained the linear regression model for dynamic viscosity y, P, as a function of mixing speed X_1, min^{-1} and mixing time X_2, min of composite rocket propellant. To determine the conditions of minimal viscosity, a method of steepest ascent has been applied. This method has defined the local optimum region that has to be described by a second-order model. Conditions of the factor variations are shown in Table 2.146.

Table 2.146 Factor variation intervals

Factors	Variation intervals					Δx
	-1.414	-1.0	0	+1.0	+1.414	
x_1-mixing speed, min^{-1}	37.72	40.00	60.00	80.00	88.28	20
x_2-mixing time, min	11.02	40.00	110.00	180.00	208.98	70

The central composite rotatable design of experiment with outcomes is shown in Table 2.147.

The reproducibility variance from five replicated design points has the value: $S_{\bar{y}}^2$=60.93 or $S_{\bar{y}}$=7.81.

The regression coefficients are determined from these relations:

$$b_0 = 0.2\sum_{1}^{13} y_u - 0.1\sum_{1}^{2}\sum_{1}^{6} X_{iu}^2 y_u = 529.12;$$

$$b_1 = 0.125\sum_{1}^{6} X_{1u} y_u = -66.40;$$

Table 2.147 CCRD $2^2 + 2x2 + 5$

No.	Design matrix		Working matrix		Response	Predicted values		
	X_1	X_2	x_1	x_2	$\bar{y}_u$	$\hat{y}_u$	$\bar{y}_u - \hat{y}_u$	$(\bar{y}_u - \hat{y}_u)^2$
1	+	+	80	180	499.2	484.7	14.50	210.26
2	−	+	40	180	640.0	637.5	2.50	6.25
3	+	−	80	40	635.2	643.5	-8.30	68.89
4	−	−	40	40	736.0	756.3	-20.30	412.09
5	-1.414	0	31.72	110	688.0	674.4	13.60	184.96
6	+1.414	0	88.28	110	483.2	486.6	-3.40	11.56
7	0	-1.414	60	11.02	800.0	778.0	21.40	457.96
8	0	+1.414	60	208.98	571.2	582.0	-11.00	121.00
9	0	0	60	110	528.0	529.12	-1.12	1.23
10	0	0	60	110	540.8	529.12	11.68	136.66
11	0	0	60	110	520.0	529.12	-9.12	82.99
12	0	0	60	110	531.2	529.12	2.08	4.37
13	0	0	60	110	524.8	529.12	-4.32	18.58
								1716.80

$$b_2 = 0.125 \sum_{1}^{6} X_{2u} y_u = -69.44;$$

$$b_{12} = 0.25 \sum_{1}^{6} X_{1u} X_{2u} y_u = -10.00;$$

$$b_{11} = 0.125 \sum_{1}^{6} X_{1u}^2 y_u + 0.0187 \sum_{1}^{2} \sum_{1}^{6} X_{iu}^2 y_u - 0.1 \sum_{1}^{13} y_u = 25.70;$$

$$b_{22} = 0.125 \sum_{1}^{6} X_{2u}^2 y_u + 0.0187 \sum_{1}^{2} \sum_{1}^{6} X_{iu}^2 y_u - 0.1 \sum_{1}^{13} y_u = 75.67;$$

The significance check of regression coefficients:

$$S_{b_0}^2 = 0.2 S_{\bar{y}}^2 = 12.19;\ S_{b_0} = 3.49;\ \Delta b_0 = \pm 2 S_{b_0} = \pm 6.98;$$

$$S_{b_i}^2 = 0.125 S_{\bar{y}}^2 = 7.62;\ S_{b_i} = 2.76;\ \Delta b_i = \pm 2 S_{b_i} = \pm 5.52;$$

$$S_{b_{ii}}^2 = 0.1438 S_{\bar{y}}^2 = 8.76;\ S_{b_{ii}} = 2.96;\ \Delta b_{ii} = \pm 2 S_{b_{ii}} = \pm 5.92;$$

$$S_{b_{ij}}^2 = 0.250 S_{\bar{y}}^2 = 15.24;\ S_{b_{ij}} = 3.90;\ \Delta b_{ij} = \pm 2 S_{b_{ij}} = \pm 7.80.$$

All regression coefficients are significant with 95% confidence. The second-order regression model has the form:

$$\hat{y}_u = 529.12 - 66.40 X_1 - 69.44 X_2 - 10.00 X_1 X_2 + 25.70 X_1^2 + 75.67 X_2^2 \qquad (2.100)$$

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Example 2.48 [39]

To optimize the process of isomerization of sulphanylamide from Problem 2.6, a screening experiment has been done by the random balance method. Factors X_1, X_2 and X_3 have been selected for this experiment. Optimization of the process is done by the given three factors at fixed values of other factors. To obtain the second-order model, a central composite rotatable design has been set up. Factor-variation levels are shown in Table 2.148. The design of the experiment and the outcomes of design points are in Table 2.149.

Table 2.148 Factor variation intervals

Factors	Variation levels				
	-1.682	**-1**	**0**	**1**	**1.682**
x_1	0.2	0.3	0.5	0.7	0.8
x_2	150	154	160	166	170
x_3	30	40	55	70	80

Table 2.149 CCRD $2^3 + 2 \times 3 + 6$

No.	Design matrix			Response	No.	Design matrix			Response
	X_1	X_2	X_3	y_u		X_1	X_2	X_3	y_u
1	+	+	+	76.00	**11**	0	-1.682	0	71.87
2	+	−	+	74.05	**12**	0	+1.682	0	77.82
3	−	−	+	80.90	**13**	0	0	-1.682	72.26
4	−	−	−	73.00	**14**	0	0	+1.682	79.07
5	+	+	−	76.81	**15**	0	0	0	77.30
6	+	−	−	62.65	**16**	0	0	0	72.80
7	−	+	−	81.40	**17**	0	0	0	77.90
8	−	+	+	82.40	**18**	0	0	0	78.40
9	-1.682	0	0	84.75	**19**	0	0	0	77.30
10	+1.682	0	0	72.42	**20**	0	0	0	77.70

An adequate regression model has been obtained by statistical processing of outcomes:

$$\widehat{y}_u = 76.89 - 2.58X_1 + 2.64X_2 + 2.26X_3 + 0.78X_1X_2 + 0.21X_1X_3 - 2.39X_2X_3 + 0.52X_1^2 - 0.81X_2^2 - 0.51X_3^2 \quad (2.101)$$

Example 2.49 [16]

A second step optimization of synthesis of methacrylic acid has been done in lab conditions. Optimization has been performed by these factors: x_1-temperature, °C; x_2-concentration of outlet α-oxy iso fatty acid in water solution, % and x_3-volume

flow of solution, l/h. Two optimization parameters have been analyzed: y_1-methacrylic acid yield from inserted α-oxy iso fatty acid and y_2-methacrylic yield as compared with dissolved α-oxy iso fatty acid (both responses are in per cent from theoretical yield). The basic experiment has been done according to *FUFE* design, as shown in Table 2.150.

Table 2.150 FUFE 2^3

Factors	x_1	x_2	x_3	y_1	y_2
+	270	30	0.6	–	–
0	255	20	0.5	82.1	85.4
–	240	10	0.4	–	–
No.	X_1	X_2	X_3		
1	–	–	–	53.0	61.5
2	+	–	–	65.3	69.0
3	–	+	–	76.1	87.6
4	+	+	–	77.0	84.5
5	–	–	+	72.7	72.7
6	+	–	+	56.1	70.5
7	–	+	+	81.0	82.7
8	+	+	+	74.0	87.0

Table 2.151 Additional design points for CCRD $2^3 + 2 \times 3 + 6$

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No.	X_1	X_2	X_3	y_1	y_2
1	-1.682	0	0	70.0	78.1
2	+1.682	0	0	72.1	79.3
3	0	-1.682	0	49.1	49.2
4	0	+1.682	0	74.8	81.4
5	0	0	-1.682	79.7	81.2
6	0	0	+1.682	83.5	90.6
7	0	0	0	82.0	86.8
8	0	0	0	82.9	86.2
9	0	0	0	83.6	84.9
10	0	0	0	82.6	85.9
11	0	0	0	83.1	86.6

Statistical data analysis has offered these regression coefficient values:

for y_1: for y_2:
b_0=69.41 b_1=-1.26; b_2=7.62; b_3=1.56; b_0=76.92; b_1=0.81; b_2=8.50; b_3=1.28;
b_{12}=-0.21; b_{13}=-4.70; b_{23}=-0.94; b_{123}=2.60. b_{12}=-0.51; b_{13}=-0.28; b_{23}=-1.88; b_{123}=2.13.

To calculate the variance of reproducibility, all design points have been replicated once. The sequence of doing the design points by *FUFE* design has been completely random. A check of significance of regression coefficients has shown that, apart from linear, interaction regression coefficients are also significant. The difference between associated free members (b_0) and response in the design center: Δy_1=82.1-69.4=12.7; Δy_2=85.4-76.9=8.5, indicates that *FUFE* has been set in factor space with a high curvature of the response surface. *FUFE* has therefore been upgraded to a second-order rotatable design, with additional design points, as shown in Table 2.151.

One design point (6-th) in the design center is missing from the additional design points. This design point is in the *FUFE* design matrix.

These regression models were obtained by statistical analysis:

$$\begin{aligned}\widehat{y}_1 &= 83.70 - 0.49X_1 + 7.63X_2 + 1.38X_3 - 0.21X_1X_2 \\ &\quad -4.71X_1X_3 - 0.94X_2X_3 - 4.41X_1^2 - 7.60X_2^2 - 0.50X_3^2\end{aligned} \tag{2.102}$$

$$\begin{aligned}\widehat{y}_2 &= 87.20 + 0.64X_1 + 8.25X_2 + 1.89X_3 - 0.51X_1X_2 \\ &\quad -0.28X_1X_3 - 1.88X_2X_3 - 2.40X_1^2 - 7.42X_2^2 - 0.24X_3^2\end{aligned} \tag{2.103}$$

Example 2.50 [16]

A process having properties dependent on four factors has been tested. A full factorial experiment and optimization by the method of steepest ascent have brought about the experiment in factor space where only two factors are significant and where an inadequate linear model has been obtained. To analyze the given factor space in detail, a central composite rotatable design has been set up, as shown in Table 2.152.

Table 2.152 CCRD $2^2 + 2 \times 2 + 5$

Variation levels	Factors		Regression coefficients
	x_1	x_2	b_0=85.14; b_1=3.43; b_2=-1.32;
Null level	9.20	4.89	b_{12}=3.00; b_{11}=2.60; b_{22}=-1.19;
Upper level	10.00	6.89	
Lower level	8.40	2.89	$\widehat{y}_u = 85,14 + 3,43X_1 - 1,32X_2 + 3,00X_1X_2 + 2,60X_1^2 - 1,19X_2^2$
+1.41	10.33	7.71	
-1.41	8.07	2.07	

	Design matrix						Response		
No.	X_0	X_1	X_2	X_1^2	X_2^2	X_1X_2	y_u	$\widehat{y}_u$	$\left(y_u - \widehat{y}_u\right)^2$
1	+	−	−	+	+	+	87.1	87.44	0.1156
2	+	−	+	+	+	−	79.0	78.80	0.0400
3	+	+	−	+	+	−	88.9	88.30	0.3600
4	+	+	+	+	+	+	92.8	91.66	1.2986
5	+	-1.41	0	2.0	0	+	85.6	85.50	0.0100
6	+	+1.41	0	2.0	0	0	94.0	95.18	1.3924
7	+	0	-1.41	0	2.0	0	84.5	84.62	0.0144
8	+	0	+1.41	0	2.0	0	80.0	80.90	0.8100
9	+	0	0	0	0	0	83.7	85.14	2.0736
10	+	0	0	0	0	0	86.0	85.14	0.7386
11	+	0	0	0	0	0	85.8	85.14	0.4356
12	+	0	0	0	0	0	83.9	85.14	1.5376
13	+	0	0	0	0	0	86.3	85.14	1.3456

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■ **Problem 2.21 [40]**

When researching the process of zirconium extraction by tributylphosphate from nitric acid solution, two factors, X_1 and X_2 have been analyzed. The observed factors and response are shown by these relations:

$$X_1 = \log_2 X_H - 1.5; X_2 = 2(\log_2 T + 2.5); y = \log_2 D,$$

where:

X_H is balancing concentration of hydrogen ion;
T is concentration of free tributylphosphate;
D is coefficient of separation.

Previous research has indicated that for mathematical modeling of the process it is necessary to use second-order rotatable design. The design matrix with experimental outcomes are given in Table 2.153.

Table 2.153 CCRD $2^2 + 2 \times 2 + 4$

No.	X_0	X_1	X_2	X_1^2	X_2^2	X_1X_2	y_{u1}	y_{u2}	y_{u3}
1	+	−	−	+	+	+	-7.94520	-7.95220	-7.94870
2	+	−	+	+	+	−	-6.25480	-6.56050	-6.40770
3	+	+	−	+	+	−	-0.29170	-0.21430	-0.25300
4	+	+	+	+	+	+	+1.36740	+1.21430	+1.29085
5	+	-1.41	0	+2	0	0	-7.18830	-7.76480	-7.47665
6	+	+1.41	0	+2	0	0	+2.05320	+2.05680	+2.05500
7	+	0	-1.41	0	+2	0	-5.28840	-5.23290	-5.26065
8	+	0	+1.41	0	+2	0	-3.05910	-3.03520	-3.07715
9	+	0	0	0	0	0	-4.33690	-4.19000	-4.26345
10	+	0	0	0	0	0	-4.18740	-4.28800	-4.23770
11	+	0	0	0	0	0	-4.19530	-4.19270	-4.19400
12	+	0	0	0	0	0	-4.19530	-4.19270	-4.19400

Problem 2.22 [41]

The research subject in the given problem is the process of cementation based on squeezing out mercury from salt-acidic solution by means of a less useful metal, such as aluminum. A study of kinetics of the given chemical reaction shows that this process may be effectively conducted in a continuous chemical reactor. Process efficiency is measured by mercury concentration in the solution after refinement. This is simultaneously the system response as it may be measured quite accurately and quantitatively. These three factors influence the cementation process significantly: X_1-temperature of solution, °C; X_2-solution flow rate in reactor, ml/l and X_3-quantity of aluminum g. The factor space is defined by these intervals: $50<X_1<100$; $300<X_2<3000$; $6<X_3<16$.

These values have been chosen for the design center and variation intervals:

$x_{10}=80$; $\Delta x_1=10$; $x_{20}=750$; $\Delta x_2=300$; $x_{30}=12.66$; $\Delta x_3=2$.

Values for all variation levels are shown in Table 2.154. Select *FUFE* 2^3 as a basic design of experiment. Determine the linear regression model from experimental outcomes, Table 2.155. Assume that the obtained linear model is inadequate and that there is curvature of the response surface. To check these assumptions, additional design points were done in the experimental center so that their average is $\bar{y}_0=0.1097$ ($\bar{y}_0$ − estimate of free member in linear regression, i.e. $\bar{y}_0 \rightarrow \beta_0$). Since $b_0 - \bar{y}_0 = \sum \beta_{ii}$ is the measure of the response surface curvature, check the value of the mentioned difference. To model the curved response surface, upgrade *FUFE* to *CCRD* and determine regression coefficients of the second-order model. The design point outcomes are in Table 2.155.

Table 2.154 Factor-variation intervals

Factors	Variation levels					Δx
	-1.682	–	0	+	+1.682	
x_1-temperature of solution	69.18	70	80	90	96.82	10
x_2-flow of solution	145.4	450	750	1050	1254.6	300
x_3-quantity of aluminum	9.3	10.66	12.66	14.66	16.02	2

Table 2.155 CCRD $2^3 + 2 \times 3 + 6$

No.	X_0	Design matrix			Response
		X_1	X_2	X_3	y_u
1	+	+	+	+	0.1082
2	+	–	+	+	0.2940
3	+	+	–	+	0.0956
4	+	–	–	+	0.1034
5	+	+	+	–	0.3855
6	+	–	+	–	0.7045
7	+	+	–	–	0.2761
8	+	–	–	–	0.4271
9	+	+1.682	0	0	0.0783
10	+	-1.682	0	0	0.3464
11	+	0	+1.682	0	0.3321
12	+	0	-1.682	0	0.0714
13	+	0	0	+1.682	0.2094
14	+	0	0	-1.682	0.7048
15	+	0	0	0	0.1224
16	+	0	0	0	0.1382
17	+	0	0	0	0.1204
18	+	0	0	0	0.0943
19	+	0	0	0	0.0698
20	+	0	0	0	0.1135

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Problem 2.23 [42]

In the study of a sort of textile fabric, its permeability to air has been tested. The research objective was to obtain a mathematical model for air permeability:

$y = f(X_1, X_2, X_3)$

where:

y l/m^2s is air permeability through textile fabric;
X_1 g/m^2 is textile fabric homogeneity;
X_2 g/cm^3 is volume weight of material;
X_3 mm W.C. is air pressure drop.

The experiment has been done by *CCRD* design. Conditions of factors variations are given in Table 2.156, and experimental outcomes in Table 2.157. Determine regression coefficients in the second-order model.

Table 2.156 Factor variation intervals

Factors	Variation levels					Δx
	-1.68	-1.00	0	+1.00	+1.68	
x_1-permeability	92	120	160	200	227	40
x_2-homogeneity	0.10	0.18	0.30	0.42	0.50	0.12
x_3-pressure drop	2	4	7	10	12	3

Table 2.157 CCRD $2^3 + 2 \times 3 + 6$

No.	Design matrix			y	No.	Design matrix			y
	X_1	X_2	X_3			X_1	X_2	X_3	
1	+	+	+	151	11	0	-1.68	0	844
2	+	+	–	70	12	0	+1.68	0	180
3	+	–	+	626	13	0	0	-1.68	140
4	+	–	–	330	14	0	0	+1.68	576
5	–	+	+	507	15	0	0	0	369
6	–	+	–	250	16	0	0	0	352
7	–	–	+	1000	17	0	0	0	354
8	–	–	–	540	18	0	0	0	335
9	-1.68	0	0	740	19	0	0	0	357
10	+1.68	0	0	227	20	0	0	0	345

■ **Problem 2.24 [43]**

The problem in this study consisted of obtaining experimental results on the kinetics of the drying process of granulated silicon dioxide in a mathematical process modeling. The drying process of solid capillary porous materials is theoretically the most studied one. Of course, by keeping in mind the condition that coefficients of heat transfer are constant. Data from literature on granule drying with 80% to 5% moisture, however, indicate considerable changes in transfer coefficients.

By taking into consideration the changes in heat transfer coefficients, we get nonlinear models whose solutions are either complex or impossible. Due to the mentioned difficulties in analysis of simultaneous processes of heat and mass transfers, mathematical modeling of the process was done by application of experimental and statistical methods. Testing the drying process in a fluidized bed of moist granules SiO_2, was done on lab equipment. These system factors were analyzed: X_1-diameter of granules, mm; X_2-length of granules, mm; X_3-air flow velocity, m/s; X_4-moisture of granules, % and X_5-air temperature, °C. The design matrix with outcomes of design points is shown in Table 2.158. Experimental results have been obtained in this way: SiO_2 granules of corresponding moisture were fed into a fluidized column, and then hot air was passed through the formed granule bed at a certain velocity. Granule samples were taken periodically during the drying process to determine moisture. From results of such measurements moisture-time diagrams were constructed. Current moisture and drying speed in the analyzed point have been determined from the diagram for each designed point. Drying speed has been exactly the response of the experiment. Determine regression coefficients in the second-order mathematical model.

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Table 2.158 CCRD $2^5 + 25 + 10$

No. trials	Design matrix					Operational matrix					Drying speed
	X_1	X_2	X_3	X_4	X_5	x_1	x_2	x_3	x_4	x_5	y_u
1	+	+	+	+	+	5	8	3	55	289	0.390
2	−	+	+	+	+	3	8	3	55	289	0.400
3	+	−	+	+	+	5	4	3	55	289	0.358
4	−	−	+	+	+	3	4	3	55	289	0.503
5	+	+	−	+	+	5	8	2.5	55	289	0.075
6	−	+	−	+	+	3	8	2.5	55	289	0.258
7	+	−	−	+	+	5	4	2.5	55	289	0.308
8	−	−	−	+	+	3	4	2.5	55	289	0.260
9	+	+	+	−	+	5	8	3	35	289	0.508
10	−	+	+	−	+	3	8	3	35	289	0.840

Table 2.158 (continued)

No. trials	Design matrix					Operational matrix					Drying speed
	X_1	X_2	X_3	X_4	X_5	x_1	x_2	x_3	x_4	x_5	y_u
11	+	−	+	−	+	5	4	3	35	289	0.580
12	−	−	+	−	+	3	4	3	35	289	0.790
13	+	+	−	−	+	5	8	2.5	35	289	0.458
14	−	+	−	−	+	3	8	2.5	35	289	0.600
15	+	−	−	−	+	5	4	2.5	35	289	0.450
16	−	−	−	−	+	3	4	2.5	35	289	0.500
17	+	+	+	+	−	5	8	3	55	189	0.240
18	−	+	+	+	−	3	8	3	55	189	0.266
19	+	−	+	+	−	5	4	3	55	189	0.233
20	−	−	+	+	−	3	4	3	55	189	0.265
21	+	+	−	+	−	5	8	2.5	55	189	0.540
22	−	+	−	+	−	3	8	2.5	55	189	0.208
23	+	−	−	+	−	5	4	2.5	55	189	0.158
24	−	−	−	+	−	3	4	2.5	55	189	0.202
25	+	+	+	−	−	5	8	3	35	189	0.402
26	−	+	+	−	−	3	8	3	35	189	0.400
27	+	−	+	−	−	5	4	3	35	189	0.325
28	−	−	+	−	−	3	4	3	35	189	0.475
29	+	+	−	−	−	5	8	2.5	35	189	0.500
30	−	+	−	−	−	3	8	2.5	35	189	0.458
31	+	−	−	−	−	5	4	2.5	35	189	0.533
32	−	−	−	−	−	3	4	2.5	35	189	0.400
33	+2.3	0	0	0	0	6.378	6	2.75	45	239	0.400
34	-2.3	0	0	0	0	1.622	6	2.75	45	239	0.600
35	0	+2.3	0	0	0	4	10.76	2.75	45	239	0.386
36	0	-2.3	0	0	0	4	1.24	2.75	45	239	0.475
37	0	0	+2.3	0	0	4	6	3.345	45	239	0.466
38	0	0	-2.3	0	0	4	6	2.155	45	239	0.333
39	0	0	0	+2.3	0	4	6	2.75	68.78	239	0.116
40	0	0	0	-2.3	0	4	6	2.75	21.22	239	0.710
41	0	0	0	0	+2.3	4	6	2.75	45	358	0.358
42	0	0	0	0	-2.3	4	6	2.75	45	120	0.375
43	0	0	0	0	0	4	6	2.75	45	239	0.316
44	0	0	0	0	0	4	6	2.75	45	239	0.183
45	0	0	0	0	0	4	6	2.75	45	239	0.235
46	0	0	0	0	0	4	6	2.75	45	239	0.291
47	0	0	0	0	0	4	6	2.75	45	239	0.325
48	0	0	0	0	0	4	6	2.75	45	239	0.361
49	0	0	0	0	0	4	6	2.75	45	239	0.359
50	0	0	0	0	0	4	6	2.75	45	239	0.230
51	0	0	0	0	0	4	6	2.75	45	239	0.361
52	0	0	0	0	0	4	6	2.75	45	239	0.350

■ **Problem 2.25 [43]**

To calculate the height of a SiO_2 drying apparatus or fluidized column and to estimate the intensity of mass and heat exchange, it is necessary to know the volume of the fluidized granule bed in operational state, i.e. fluidized state. Based on literature data about fluidization with gases at increased velocity of gas flow, a bed of fluidized granules passes from calm homogeneous into inhomogeneous fluidization. There is no quantitative dependence, of satisfactory accuracy, about spreading of the fluidized bed. Therefore the objective of this research is a study of spreading a SiO_2 fluidized granule bed with an elaboration of a mathematical model of the process. Prior studies have shown that the process may be mathematically described only in this temperature interval: 250 – 400 °C. Process factors are: X_1-air temperature, °C; X_2-air velocity (taken at 20 °C), m/s; X_3-diameter of moist granules, mm and X_4-moisture of granules, %. The design of the experiment with outcomes is shown in Table 2.160.

Table 2.160 CCRD $2^4 + 2 \times 4 + 7$

No. trials	Sequence of trials	Design matrix				Operational matrix				y=H/H$_0$
		X_1	X_2	X_3	X_4	x_1	x_2	x_3	x_4	
1	16	–	–	–	–	250	2.5	3	40	5.6
2	11	+	–	–	–	350	2.5	3	40	8.8
3	8	–	+	–	–	250	3	3	40	7.8
4	24	+	+	–	–	350	3	3	40	11.0
5	28	–	–	+	–	250	2.5	5	40	3.2
6	7	+	–	+	–	350	2.5	5	40	6.12
7	25	–	+	+	–	250	3	5	40	6.0
8	12	+	+	+	–	350	3	5	40	7.8
9	2	–	–	–	+	250	2.5	3	60	3.3
10	4	+	–	–	+	350	2.5	3	60	3.7
11	6	–	+	–	+	250	3	3	60	6.7
12	15	+	+	–	+	350	3	3	60	4.9
13	18	–	–	+	+	250	2.5	5	60	2.4
14	23	+	–	+	+	350	2.5	5	60	3.0
15	20	–	+	+	+	250	3	5	60	3.9
16	24	+	+	+	+	350	3	5	60	3.5
17	3	-2	0	0	0	200	2.75	4	50	3.27
18	1	+2	0	0	0	400	2.75	4	50	5.1
19	17	0	-2	0	0	300	2.25	4	50	3.4
20	10	0	+2	0	0	300	3.25	4	50	7.75
21	14	0	0	-2	0	300	2.75	2	50	9.37
22	21	0	0	+2	0	300	2.75	6	50	4.5
23	31	0	0	0	-2	300	2.75	4	30	9.18
24	26	0	0	0	+2	300	2.75	4	70	2.8

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Table 2.160 (continued)

No. trials	Sequence of trials	Design matrix				Operational matrix				$y=H/H_0$
		X_1	X_2	X_3	X_4	x_1	x_2	x_3	x_4	
25	9	0	0	0	0	300	2.75	4	50	4.4
26	22	0	0	0	0	300	2.75	4	50	3.8
27	19	0	0	0	0	300	2.75	4	50	3.7
28	30	0	0	0	0	300	2.75	4	50	4.0
29	5	0	0	0	0	300	2.75	4	50	4.4
30	13	0	0	0	0	300	2.75	4	50	4.0
31	29	0	0	0	0	300	2.75	4	50	4.5

Problem 2.26 [44]

Mathematical designing of an experiment has been applied to mathematical modeling of solidification and hardness of concrete as a function of three basic factors: X_1-cement consumption, kg/m^3; X_2-percentage of sand in filler mixture, % and X_3-water consumption, l/min. This parameter was measured as response: y_1-concrete solidification, s. The cement of the same brand and the sand from the same supplier have been used in all design points. A mixture 10 l in volume was mixed manually for 3 min and a 7 l volume for 2.5 min. Samples of $10 \times 10 \times 10$ cm were prepared on a vibration table with amplitude of 0.45–0.50 mm, frequency of 2800 min^{-1} and under pressure of 80-100 kp/cm^2. Concrete solidification was measured 10–15 min after formation of samples by GOST 10181-62. Basic experiment was done by *FUFE* 2^3, as shown in Table 2.161.

Table 2.161 FUFE 2^3

Variation levels	Design matrix			Response
	x_1	x_2	x_3	
Basic level	525	0.31	189	
Variation interval	75	0.09	21	
Upper level	600	0.4	210	
Lower level	450	0.22	168	
Number of design points	X_1	X_2	X_3	Y
1	–	–	–	160
2	–	–	+	66
3	–	+	–	58
4	–	+	+	8
5	+	–	–	225
6	+	–	+	42
7	+	+	–	160
8	+	+	+	23

According to the preliminary information, concrete solidification is the nonlinear function of the composition. Prove it by processing *FUFE* outcomes. To check the estimates of the sum of coefficients next to square members, six design points in the experimental center were done and these outcomes obtained: 45; 49; 44; 40; 42 and 44. Assert the hypothesis that regression coefficients next to square members differ from zero by comparing the difference $b_0 - \bar{y}_u$ with the experimental error. This means that, to obtain a second-order model by *CCRD*, it was necessary to do another six design points in starlike points. The outcomes of all 20 design-points are given in Table 2.162. Determine the second-order model.

Table 2.162 CCRD $2^3 + 2 \times 3 + 6$

X_1	X_2	X_3	X_1^2	X_2^2	X_3^2	X_1X_2	X_1X_3	X_2X_3	y
-	–	–	+	+	+	+	+	+	160
-	–	+	+	+	+	+	–	–	66
-	+	–	+	+	+	–	+	–	58
-	+	+	+	+	+	–	–	+	8
+	–	–	+	+	+	–	–	+	225
+	–	+	+	+	+	–	+	–	42
+	+	–	+	+	+	+	–	–	160
+	+	+	+	+	+	+	+	+	23
-1.682	0	0	2.828	0	0	0	0	0	35
+1.682	0	0	2.828	0	0	0	0	0	72
0	-1.682	0	0	2.828	0	0	0	0	137
0	+1.682	0	0	2.828	0	0	0	0	34
0	0	-1.682	0	0	2.828	0	0	0	261
0	0	+1.682	0	0	2.828	0	0	0	14
0	0	0	0	0	0	0	0	0	45
0	0	0	0	0	0	0	0	0	49
0	0	0	0	0	0	0	0	0	44
0	0	0	0	0	0	0	0	0	40
0	0	0	0	0	0	0	0	0	42
0	0	0	0	0	0	0	0	0	44

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■ **Problem 2.27 [45]**

In car tire development, an optimal combination of three-component composition has been researched: X_1-hydrated silicate, PHR; X_2-silan, PHR and X_3-sulfur, PHR. These parameters were measured as system responses: y_1-PICO abrasion index; y_2-200% module; y_3-strain at brake and y_4-hardness. The experiment has been done in accord with *CCRD*. Experimental outcomes are shown in Table 2.163. Determine regression coefficients values for all four responses.

Table 2.163 CCRD $2^3 + 2 \times 3 + 6$

ariation levels	Design matrix			Responses			
	x_1	x_2	x_3				
0	1.2	50	2.3				
Δx	0.5	10	0.5				
+	1.7	60	2.8				
-	1.0	40	1.8				
No. trials	X_1	X_2	X_3	y_1	y_2	y_3	y_4
1	–	–	–	102	900	470	67.5
2	+	–	–	120	860	410	65
3	–	+	–	117	800	570	77.5
4	+	+	–	198	2294	240	74.5
5	–	–	+	103	490	640	62.5
6	+	–	+	132	1289	270	67
7	–	+	+	132	1270	410	78
8	+	+	+	139	1090	380	70
9	-1.68	0	0	102	770	590	76
10	+1.68	0	0	154	1690	260	70
11	0	-1.68	0	96	700	520	63
12	0	+1.68	0	163	1540	380	75
13	0	0	-1.68	116	2184	520	65
14	0	0	+1.68	153	1784	290	71
15	0	0	0	133	1300	380	70
16	0	0	0	133	1300	380	68.5
17	0	0	0	140	1145	430	68
18	0	0	0	142	1090	430	68
19	0	0	0	145	1260	390	69
20	0	0	0	142	1344	390	70

2.3.3
Orthogonal Second-order Design (Box-Benken Design)

Orthogonal designs have been used in the first few studies having to do with the application of designed experiments in obtaining regression models of the second-order. Although much more optimized designs for obtaining second-order models were presented in the previous chapter, second-order orthogonal design is still used in practice.

The expression (2.74), which defines the condition for design orthogonality in a general case, indicates in the matrix for central composite second-order design that all vector columns are not orthogonal. To provide design orthogonality and alleviate calculation of regression-equation coefficients, square variables are transformed and

suitable distances of "*starlike*" points chosen. Transformation is shown by the expression:

$$\left(X'_{iu}\right)^2 = X_{iu}^2 - \frac{\sum_1^N X_{iu}^2}{N} = X_{iu}^2 - \bar{X}_{iu}^2 \tag{2.104}$$

Thus the following condition is fulfilled:

$$\sum_1^N X_0 X'_{iu} = 0 \tag{2.105}$$

It has already been said that orthogonality of other vectors-columns provides a selection of distances of "starlike" points. The design matrix of *central composite orthogonal designs (CCOD)* is obtained at different number of factors (k), by upgrading the associated *FUFE* designs or half-replicas (for k≥5) with additional points in the experimental center and a corresponding number of "starlike" points. The number of *CCOD* design points with values of "*starlike*" distances is given in Table 2.164. Design matrices for k=2–4 are given in Tables 2.165–2.167.

Table 2.164 Construction of orthogonal designs

No. of factors k	No. of core points n_j	No. of starlike points n_α	No. of null points n_0	Coded values α	Total no. of design points N
2	4	4	1	1.000	9
3	8	6	1	1.215	15
4	16	8	1	1.414	25
5*	16	10	1	1.414	27
6*	32	12	1	–	45

* Core of half-replica design

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Table 2.165 Central composite orthogonal design $2^2 + 2 \times 2 + 1$

No. trials	Design matrix				Square factors		Operational matrix		Response
	X_0	X_1	X_2	X_1X_2	$\left(X_1'\right)^2 = X_1^2 - \frac{2}{3}$	$\left(X_2'\right)^2 = X_2^2 - \frac{2}{3}$	x_1	x_2	$\bar{y}_u$
1	+	+	+	+	0.33	0.33	2.5	120	50
2	+	−	+	−	0.33	0.33	1.3	120	67
3	+	+	−	−	0.33	0.33	2.5	20	70
4	+	−	−	+	0.33	0.33	1.3	20	60
5	+	−	0	0	0.33	-0.67	1.3	70	70
6	+	+	0	0	0.33	-0.67	2.5	70	56
7	+	0	−	0	-0.67	0.33	1.9	20	73
8	+	0	+	0	-0.67	0.33	1.9	120	60
9	+	0	0	0	-0.67	-0.67	1.9	70	62
Sum of squares	9	6	6	4	2	2			

In accord with the performed transformation, regression coefficient b_0 is determined from the expression:

$$b_0 = b_0' - b_{11}\bar{X}_1^2 - b_{22}\bar{X}_2^2 - \ldots - b_{kk}\bar{X}_k^2 \tag{2.106}$$

and a check of its significance is estimated by variance:

$$S_{b_0}^2 = S_{b_0}^2 + \bar{X}_1^2 S_{b_{ii}}^2 + \bar{X}_2^2 S_{b_{ii}}^2 + \ldots + \bar{X}_k^2 S_{b_{ii}}^2 \tag{2.107}$$

Other regression coefficients are determined in accord with formulas for orthogonal designing. A check of significance of regression coefficients is given in Sect. 2.4.1, and the check of lack of fit of regression model is in Sect. 2.4.3.

Example 2.51

This example refers to response dependence on two factors (k=2). Orthogonal second-order design in this case, according to Table 2.164, has nine design points (N=9). The design matrix with outcomes of design points is shown in Table 2.165. The same case has been elaborated in the previous section, in Example 2.43, by application of rotatable second-order design. However, the connection between coded and real values of factors for the same null point is now different:

$$X_1 = \frac{x_1 - 1.9}{0.6}; \quad X_2 = \frac{x_2 - 70}{50} \tag{2.108}$$

Table 2.166 Central composite orthogonal design $2^3 + 2 \times 3 + 1$

No. of design points	Design matrix										Response $\bar{y}_u$
	X_0	X_1	X_2	X_3	$(X_1')^2$	$(X_2')^2$	$(X_3')^2$	X_1X_2	X_1X_3	X_2X_3	
1	+	+	+	+	0.270	0.270	0.270	+	+	+	82
2	+	−	+	+	0.270	0.270	0.270	−	−	+	82
3	+	+	−	+	0.270	0.270	0.270	−	+	−	42
4	+	−	−	+	0.270	0.270	0.270	+	−	−	70
5	+	+	+	−	0.270	0.270	0.270	+	−	−	60
6	+	−	+	−	0.270	0.270	0.270	−	+	−	80
7	+	+	−	−	0.270	0.270	0.270	−	−	+	48
8	+	−	−	−	0.270	0.270	0.270	+	+	+	70
9	+	-1.215	0	0	0.747	-0.730	-0.730	0	0	0	80
10	+	+1.215	0	0	0.747	-0.730	-0.730	0	0	0	60
11	+	0	-1.215	0	-0.730	0.747	-0.730	0	0	0	54
12	+	0	+1.215	0	-0.730	0.747	-0.730	0	0	0	88
13	+	0	0	-1.215	-0.730	-0.730	0.747	0	0	0	85
14	+	0	0	+1.215	-0.730	-0.730	0.747	0	0	0	74
15	+	0	0	0	-0.730	-0.730	-0.730	0	0	0	70
Sum of squares	15	10.94	10.94	10.94	4.34	4.34	4.34	8	8	8	

This may be explained by the necessity of changing factor-variation intervals. Linear regression coefficients are calculated by the formula (2.62), which is:

$$b_i = \frac{\sum_{1}^{6} X_{iu}\bar{y}_u}{6} \tag{2.109}$$

so that:

$$b_1 = \frac{\sum_{1}^{6} X_{iu}\bar{y}_u}{6} = \frac{1}{6}(50 - 67 + 70 - 60 - 70 + 56) = -3.50$$

$$b_2 = \frac{\sum_{1}^{6} X_{2u}\bar{y}_u}{6} = -4.33$$

Coefficient b_{12}, which characterizes the effect of even interaction is determined in accord with the formula

$$b_{12} = \frac{\sum_{1}^{4} X_1X_2\bar{y}_u}{4} = \frac{1}{4}(50 - 67 - 70 + 60) = -6.75.$$

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To determine coefficients b_{11} and b_{12}, we use:

$$b_{ii} = \frac{\sum^{N} (X'_{iu})^2 \bar{y}_u}{\frac{1}{\sum (X'_{iu})^2}}; \tag{2.110}$$

or:

$$b_{ii} = \frac{\sum^{9} (X'_{iu})^2 \bar{y}_u}{\frac{1}{2}} \tag{2.111}$$

So that:

b_{11}=0.5[0.33(50+67+70+60+70+56)-0.67(73+60+62)]=-3.78;
b_{22}=0.5[0.33(50+67+70+60+73+60)-0.67(70+56+62)]=-0.30.

To calculate b_0 we have to determine b'_0:

$$b'_0 = \frac{\sum^{N} \bar{y}_u}{\frac{1}{N}\sum_{1}^{N} X_{iu}^2} = \frac{\sum^{N} \bar{y}_u}{\frac{1}{N}} \tag{2.112}$$

$$b'_0 = \frac{\sum^{9} \bar{y}_u}{\frac{1}{9}} = \frac{568}{9} = 63.11$$

From Eq. (2.106) we get:

$$b_0 = b'_0 - b_{11}\bar{X}_1^2 - b_{22}\bar{X}_2^2 \tag{2.113}$$

where:

$$\bar{X}_1^2 = \frac{\sum^{N} X_1^2}{\frac{1}{N}} = \frac{\sum^{9} X_1^2}{\frac{1}{9}} = \frac{2}{3}$$

so that: b_0=b'_0-0.67b_{11}-0.67b_{22}=65.84

A check of statistical significance of regression coefficients (Sect. 2.4.2), indicates that regression coefficients b_{11} and b_{22} are statistically insignificant. The final form of the second-order regression model with 95% confidence may be given in the form:

$$\hat{y}_u = 65.84 - 3.50\,X_1 - 4.33\,X_2 - 6.75\,X_1X_2 \tag{2.114}$$

Lack of fit of the obtained regression model is checked by formulas from Sect. 2.4.3 for the variant of an identical number of replications of all the design points (n=25). The obtained outcomes are given in Example 2.63 and Example 2.65.

Example 2.52

The second-order orthogonal design for k=3, is shown in Table 2.166. Based on design-point outcomes, we can calculate regression coefficients:

$$b_i = \frac{\sum_{1}^{10} X_{iu}\bar{y}_u}{10.94}; \quad b_{ij} = \frac{\sum_{1}^{8} X_{iu}X_{ju}\bar{y}_u}{8};$$

$$b_{ii} = \frac{\sum_{1}^{15} \bar{X}_{iu}^2\bar{y}_u}{4.34}; \quad b_0' = \frac{\sum_{1}^{15} \bar{y}_u}{15};$$

$$b_0 = b_0' - 0.73b_{11} - 0.73b_{22} - 0.73b_{33}.$$

After processing the outcomes, we obtain the regression model:

$$\hat{y}_u = 75.6 - 8.6X_1 + 10.5X_2 + 0.4X_3 - 5.1X_1^2 - 4.4X_2^2 + 1.3X_3^2 + 3.7X_1X_2 + 1.7X_1X_3 + 3.7X_2X_3$$

The obtained regression model is adequate with 95% confidence. A check of significance of regression coefficients, in accord with Sect. 2.4.2, is completed thus:

$$S_{b_0}^2 = \frac{S_{\bar{y}}^2}{15}; \quad S_{b_i}^2 = \frac{S_{\bar{y}}^2}{10.94}; \quad S_{b_{ij}}^2 = \frac{S_{\bar{y}}^2}{8}; \quad S_{b_{ii}}^2 = \frac{S_{\bar{y}}^2}{4.34};$$

If $S_{\bar{y}}^2 = 15$, then:

$$S_{b_0}^2 = 1.00; \quad S_{b_i}^2 = 1.36; \quad S_{b_{ij}}^2 = 1.87; \quad S_{b_{ii}}^2 = 3.45.$$

Then:
$\Delta b_0 = \pm 2.0$; $\Delta b_i = \pm 2.34$; $\Delta b_{ij} = \pm 2.74$; $\Delta b_{ii} = \pm 3.70$.
With 95% confidence then:

$$\hat{y}_u = 75.6 - 8.61X_1 + 10.5X_2 - 5.1X_1^2 - 4.4X_2^2 + 3.7X_1X_2 + 3.7X_2X_3 \qquad (2.115)$$

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Example 2.53

The orthogonal design for four factors (k=4) is given in Table 2.167 with outcomes of design points. The regression coefficients are determined thus:

$$b_i = \frac{\sum_{1}^{18} X_{iu}\bar{y}_u}{20}; \quad b_{ij} = \frac{\sum_{1}^{16} X_{iu}X_{ju}\bar{y}_u}{16}; \quad b_{ii} = \frac{\sum_{1}^{25} X_{iu}^2\bar{y}_u}{8}; \quad b_0' = \frac{\sum_{1}^{25} \bar{y}_u}{25};$$

$$b_0 = b_0' - 0.80b_{11} - 0.80b_{22} - 0.80b_{33} - 0.80b_{44}$$

The second-order regression model has the following form:

$$\hat{y}_u = 82.8 + 18.4X_1 + 17.3X_2 - 22.6X_3 + 30.7X_4 - 6.9X_1^2 + 1.4X_2^2 + 2.4X_3^2$$
$$-0.5X_4^2 + 1.5X_1X_2 - 5.6X_1X_3 + 6.1X_1X_4 - 1.4X_2X_3 + 5.3X_2X_4 - 7.0X_3X_4 \qquad (2.116)$$

Table 2.167 Central composite orthogonal design $2^4 + 2 \times 4 + 1$

No. trials	Design matrix										Response	Predicted response
	X_0	X_1	X_2	X_3	X_4	$(X_1')^2$	$(X_2')^2$	$(X_3')^2$	$(X_4')^2$	X_1X_2	$\bar{y}_u$	$\hat{y}_u$
1	+	+	+	+	+	0.2	0.2	0.2	0.2	+	118	122
2	+	−	+	+	+	0.2	0.2	0.2	0.2	−	80	81
3	+	+	−	+	+	0.2	0.2	0.2	0.2	−	78	76
4	+	−	−	+	+	0.2	0.2	0.2	0.2	+	42	42
5	+	+	+	−	+	0.2	0.2	0.2	0.2	+	200	195
6	+	−	+	−	+	0.2	0.2	0.2	0.2	−	128	132
7	+	+	−	−	+	0.2	0.2	0.2	0.2	−	145	144
8	+	−	−	−	+	0.2	0.2	0.2	0.2	+	88	87
9	+	+	+	+	−	0.2	0.2	0.2	0.2	+	52	52
10	+	−	+	+	−	0.2	0.2	0.2	0.2	−	33	35
11	+	+	−	+	−	0.2	0.2	0.2	0.2	−	30	28
12	+	−	−	+	−	0.2	0.2	0.2	0.2	+	14	17
13	+	+	+	−	−	0.2	0.2	0.2	0.2	+	95	97
14	+	−	+	−	−	0.2	0.2	0.2	0.2	−	58	58
15	+	+	−	−	−	0.2	0.2	0.2	0.2	−	70	67
16	+	−	−	−	−	0.2	0.2	0.2	0.2	+	37	34
17	+	-1.414	0	0	0	1.2	-0.8	-0.8	-0.8	0	48	43
18	+	+1.414	0	0	0	1.2	-0.8	-0.8	-0.8	0	90	95
19	+	0	-1.414	0	0	-0.8	1.2	-0.8	-0.8	0	55	61
20	+	0	+1.414	0	0	-0.8	1.2	-0.8	-0.8	0	116	110
21	+	0	0	-1.414	0	-0.8	-0.8	1.2	-0.8	0	115	120
22	+	0	0	+1.414	0	-0.8	-0.8	1.2	-0.8	0	60	56
23	+	0	0	0	-1.414	-0.8	-0.8	-0.8	1.2	0	38	38
24	+	0	0	0	+1.414	-0.8	-0.8	-0.8	1.2	0	125	125
25	+	0	0	0	0	-0.8	-0.8	-0.8	-0.8	0	84	83
Sum of squares	25	20	20	20	20	8	8	8	8	16		

A check of lack of fit of the regression model (2.116), is done in accordance with the formulas from Sect. 2.4.3, where all design points are replicated the same number of times (n=25). The obtained predicted response values of the regression model are also given in Table 2.167. Variance of lack of fit is calculated thus:

$$S_{AD}^2 = \frac{\sum_1^{25} n\left(\bar{y}_u - \hat{y}_u\right)^2}{N - \frac{(k+2)(k+1)}{2}} = \frac{25 \times 267}{25 - 15} = 667.5$$

The arithmetic value of Fisher's criterion is determined for S_y^2=375.0.

$$F_R = \frac{667.5}{375.0} = 1.78$$

A tabular value $F_{T.}$=1.85 is obtained for f_{AD}=10 and f_E=25(25-1)=600 from Table E. The regression model is with 95% confidence adequate because $F_R < F_{T.}$. To estimate the statistical significance of regression coefficients, use formulas from Sect. 2.4.2.

$$S^2_{b_0} = \frac{S^2_{\bar{y}}}{25}; \quad S^2_{b_i} = \frac{S^2_{\bar{y}}}{20}; \quad S^2_{b_{ij}} = \frac{S^2_{\bar{y}}}{16}; \quad S^2_{b_{ii}} = \frac{S^2_{\bar{y}}}{8}; \quad S^2_{\bar{y}} = \frac{S^2_y}{n} = 15$$

So that:

$$S^2_{b_0} = 0.60; \quad S^2_{b_i} = 0.75; \quad S^2_{b_{ij}} = 0.94; \quad S^2_{b_{ii}} = 1.87.$$

$\Delta b_0 = \pm 2 \times 0.77 = \pm 1.54$; $\Delta b_i = \pm 2 \times 0.87 = \pm 1.74$; $\Delta b_{ij} = \pm 2 \times 0.97 = \pm 1.94$; $\Delta b_{ii} = \pm 2 \times 1.37 = \pm 2.74$.

Having taken the given values into consideration, the regression model becomes:

$$\hat{y}_u = 82.8 + 18.4X_1 + 17.3X_2 - 22.6X_3 + 30.7X_4 - 6.9X_1^2 + 6.1X_1X_4 - 5.6X_1X_3 + 5.3X_2X_4 - 7.0X_3X_4 \quad (2.117)$$

Table 2.168 Sums of squares of columns

Number of factors k	Value of sum $\sum_1^N X_{iu}^2$			
	b_0	b_i	b_{ij}	b_{ii}
2	9	6	4	2
3	15	10.94	8	4.34
4	25	20	16	8
5	27	20	16	8

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To calculate regression coefficients more efficiently, sums of squares of elements in design matrix columns are shown in Table 2.168.

Example 2.54 [12]

The oxidation process of sodium hypophosphite with iron has been studied. To establish conditions of quantitative oxidation of NaH_2PO_2 with iron and the possible reaction surface, an experiment has been defined for obtaining a mathematical model of the process. The experimental objective deals with the conditions for obtaining 100% oxidation of NaH_2PO_2.

In accordance with the data from the literature, the reaction in a constant volume of 100 ml and at the boiling point temperature is defined by these three factors:

X_1 iron concentration;

X_2 acidity and

X_3 oxidation time.

All design points are done in 100 ml volume with 20.14 mg of $NaH_2PO_2H_2O$. The reaction has been interrupted by a sudden cooling of the solution, after which the quantity of oxidized hypophosphite was determined. The system response is the rate of sodium hypophosphite oxidation. The suggestion is to apply *CCOD* to obtain a second-order mathematical model. The experiment has been designed according to orthogonal design, after optimization by the steepest ascent method and with the design center in the design point, which had the greatest oxidation rate of NaH_2PO_2. The design of experiment with outcomes of design points is given in Table 2.169.

Table 2.169 CCOD $2^3 + 2 \times 3 + 1$

Variation levels		Factors				
		X_1	X_2	X_3		
Basic level		0.032	1.0	15		
Variation interval		0.005	0.5	5		
No. trials	Design matrix				Response	
	X_0	X_1	X_2	X_3	y_u	$\widehat{y}_u$
1	+	−	−	−	96.18	94.19
2	+	+	−	−	97.88	97.65
3	+	−	+	−	92.96	93.61
4	+	+	+	−	98.34	97.55
5	+	−	−	+	97.36	98.47
6	+	+	−	+	98.18	98.93
7	+	−	+	+	95.24	94.89
8	+	+	+	+	99.32	98.83
9	+	-1.215	0	0	98.30	97.02
10	+	+1.215	0	0	98.40	99.69
11	+	0	-1.215	0	99.78	97.68
12	+	0	+1.215	0	94.53	95.45
13	+	0	0	-1.215	97.34	97.57
14	+	0	0	+1.215	99.24	99.13
15	+	0	0	0	99.08	98.55

$$b_0' = \frac{\sum_{1}^{N} \bar{y}_u}{N} = \frac{1462.13}{15} = 97.48$$

$$b_1 = \frac{\sum_{1}^{10} X_{1u}\bar{y}_u}{10.94}$$

$$= \frac{1}{10.94}(-96.18 + 97.88 - 92.96 + 98.34 - 97.36 + 98.18 - 95.24 + 99.32 - 1.215 \times 98.30 - 1.215 \times 98.40) = 1.11$$

$$b_2 = \frac{\sum_{1}^{10} X_{2u}\bar{y}_u}{10.94} = \frac{1}{10.94}(-96.18-97.88+92.96+98.34-97.36-98.18 +95.24+99.32-1.21599.78+1.21594.53)=-0.92$$

$$b_3 = \frac{\sum_{1}^{10} X_{3u}\bar{y}_u}{10.94} = \frac{1}{10.94}(-96.18-97.88-92.96-98.34+97.36+98.18 +95.24+99.32-1.21597.34+1.21599.24)=0.64$$

$$b_{12} = \frac{\sum_{1}^{8} X_{1u}X_{2u}\bar{y}_u}{8} = \frac{1}{8}(96.18-97.88-92.96+98.34+97.36-98.18-95.24+99.32)=0.87$$

$$b_{13} = \frac{\sum_{1}^{8} X_{1u}X_{3u}\bar{y}_u}{8} = \frac{1}{8}(96.18-97.88+92.96-98.34-97.36+98.18-95.24+99.32)=-0.27$$

$$b_{23} = \frac{\sum_{1}^{8} X_{2u}X_{3u}\bar{y}_u}{8} = \frac{1}{8}(96.18+97.88-92.96-98.34-97.36-98.18+95.24+99.32)=0.22$$

$$b_{11} = \frac{\sum_{1}^{15} X_{1u}'\bar{y}_u}{4.34} = \frac{1}{4.34}(0.27096.18+0.27097.88+0.27092.96+0.27098.34 +0.27097.36+0.27098.18+0.27095.24+0.27099.32+0.74798.30+0.74798.40 -0.73099.78-0.73094.53-0.73097.34-0.73099.24-0.73099.08)=-0.32$$

$$b_{22} = \frac{\sum_{1}^{15} X_{2u}'\bar{y}_u}{4.34} = \frac{1}{4.34}(0.27096.18+0.27097.88+0.27092.96+0.27098.34 +0.27097.36+0.27098.18+0.27095.24+0.27099.32-0.73098.30-0.73098.40 +0.74799.78+0.74794.53-0.73097.34-0.73099.24-0.73099.08)=-1.12$$

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$$b_{33} = \frac{\sum_{1}^{15} X'_{3u}\bar{y}_u}{4.34} = \frac{1}{4.34}(0.27096.18+0.27097.88+...+0.27099.32-0.73098.30$$

$$-0.73098.40-0.73099.78-0.73094.53+0.74797.34+0.74799.24-0.73099.08)$$
$$=-0.36.$$

$$b_0 = b_0' - b_{11}\bar{X}_1^2 - b_{22}\bar{X}_2^2 - b_{33}\bar{X}_3^2 = 97.48-0.73(-0.32)-0.73(-1.12)-0.73(-0.36)=98.79$$

A check of significance of regression coefficients shows that with 95% confidence the following regression coefficients are statistically significant: b_0; b_1; b_2; b_3; b_{12} and b_{22}. The second-order regression model has this form:

$$\widehat{y}_u = 98.79+1.11X_1-0.92X_2+0.64X_3+0.87X_1X_2-1.12XX_2^2 \quad (2.118)$$

Example 2.55 [38]

The conditions for maximal dissolving of borate in a mixture of sulfurous and phosphorous acids are investigated. The rate of dissolving borate (y) is controlled by varying these factors: X_1-temperature of reaction, °C; X_2-reaction time, min; X_3-stoichiometric ratio of phosphorous acid in per cent, % and X_4-concentration of phosphorous acid as, % P_2O_5.

The basic levels and variation intervals are given in Table 2.170. It is known from previous design points that the optimum is within the studied factor space. Orthogonal design has therefore been done to obtain the regression model, Table 2.171. Reproducibility variance of the experiment is determined from four additional design points in the experimental center (y_{01}=61.8; y_{02}=59.3; y_{03}=58.7 and y_{04}=69.0).

Table 2.170 Factor-variation intervals

Factors	Variation levels					Variation intervals
	-1.414	-1.0	0	+1.0	+1.414	Δx
x_1-temperature	19.75	30	55	80	90.25	25
x_2-time	5.78	15	37.5	60	69.23	22.5
x_3-stechiometric ratio	51.80	60	80	100	108.20	20
x_4-concentration of H_3PO_4	6.29	14	32.8	51.60	59.31	18.8

$$\bar{y}_0 = \frac{\sum_{1}^{4} y_{0i}}{4} = 60.95; \quad S_{\bar{y}}^2 = \frac{\sum_{1}^{n_0} (y_{0i}-\bar{y}_0)^2}{n_0-1} = 5.95; \quad f = 4-1 = 3.$$

Regression coefficients have the values:

b_0'=61.54 b_1=17.37 b_2=6.4 b_3=4.7 b_4=-4.37 b_{44}=-5.34
b_{12}=2.18 b_{13}=0.2 b_{14}=1.2 b_{23}=0.56 b_{24}=0.79 b_{11}=4.5
b_{22}=1.3 b_{33}=4.09.

After rejecting statistically insignificant regression coefficients, the regression model becomes:

$$\hat{y}=61.54+17.37X_1+6.4X_2+4.7X_3-4.37X_4+2.18X_1X_2+1.9X_3X_4+4.5(X_1^2-0.8)$$
$$+4.09(X_3^2-0.8)-5.34(X_4^2-0.8)=58.9+17.37X_1+6.4X_2+4.7X_3-4.37X_4 \quad (2.119)$$
$$+2.18X_1X_2+1.9X_3X_4+4.5X_1^2+4.09X_3^2-5.34X_4^2$$

Table 2.171 CCOD $2^4 + 2 \times 4 + 1$

No. trials	X_0	X_1	X_2	X_3	X_4	$(X_1')^2$	$(X_2')^2$	$(X_3')^2$	$(X_4')^2$	X_1X_2	X_1X_3	X_1X_4	X_2X_3	X_2X_4	X_3X_4	y
1	+	+	+	+	+	0.2	0.2	0.2	0.2	+	+	+	+	+	+	86.9
2	+	−	+	+	+	0.2	0.2	0.2	0.2	+	−	−	−	−	+	40.0
3	+	+	+	−	+	0.2	0.2	0.2	0.2	−	−	+	+	−	−	66.0
4	+	−	+	−	+	0.2	0.2	0.2	0.2	−	+	−	−	+	−	34.4
5	+	+	+	+	−	0.2	0.2	0.2	0.2	−	+	−	−	+	−	76.8
6	+	−	+	+	−	0.2	0.2	0.2	0.2	−	−	+	+	−	−	55.7
7	+	+	+	−	−	0.2	0.2	0.2	0.2	+	−	−	−	−	+	91.0
8	+	−	+	−	−	0.2	0.2	0.2	0.2	+	+	+	+	+	+	47.6
9	+	+	−	+	+	0.2	0.2	0.2	0.2	−	+	+	−	−	+	74.1
10	+	−	−	+	+	0.2	0.2	0.2	0.2	−	−	−	+	+	+	52.0
11	+	+	−	−	+	0.2	0.2	0.2	0.2	+	−	+	−	+	−	74.5
12	+	−	−	−	+	0.2	0.2	0.2	0.2	+	+	−	+	−	−	29.6
13	+	+	−	+	−	0.2	0.2	0.2	0.2	+	+	−	+	−	−	94.8
14	+	−	−	+	−	0.2	0.2	0.2	0.2	+	−	+	−	+	−	49.6
15	+	+	−	−	−	0.2	0.2	0.2	0.2	−	−	−	+	+	+	68.6
16	+	−	−	−	−	0.2	0.2	0.2	0.2	−	+	+	−	−	+	51.8
17	+	0	0	0	0	-0.8	-0.8	-0.8	-0.8	0	0	0	0	0	0	61.8
18	+	+1.414	0	0	0	1.2	-0.8	-0.8	-0.8	0	0	0	0	0	0	95.4
19	+	-1.414	0	0	0	1.2	-0.8	-0.8	-0.8	0	0	0	0	0	0	41.7
20	+	0	+1.414	0	0	-0.8	1.2	-0.8	-0.8	0	0	0	0	0	0	79.0
21	+	0	-1.414	0	0	-0.8	1.2	-0.8	-0.8	0	0	0	0	0	0	42.4
22	+	0	0	+1.414	0	-0.8	-0.8	1.2	-0.8	0	0	0	0	0	0	77.6
23	+	0	0	-1.414	0	-0.8	-0.8	1.2	-0.8	0	0	0	0	0	0	58.0
24	+	0	0	0	+1.414	-0.8	-0.8	-0.8	1.2	0	0	0	0	0	0	45.6
25	+	0	0	0	-1.414	-0.8	-0.8	-0.8	1.2	0	0	0	0	0	0	52.3

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Example 2.56 [21]

This example refers to the research of this chemical reaction:

$$A+B \rightarrow C+D$$

where two reactants, A and B, give the mixture of product C and D. The final product of the chemical reaction gives mixture C and D and unchanged components A and B. The experimental objective is to get maximal yield of components C, under the condition that D's content does not exceed 20% (more than 20% of the component D causes difficulties in refining). The research included variations of these factors: X_1-temperature, °C; X_2-initial concentration of reactant A, %; X_3-reaction time, h. The contents of component B have been maintained constant. Prior research of this chemical reaction indicates the existence of interactions between the analyzed factors. Therefore, *FUFE* 2^3 with the following factor-variation levels was selected as the basic design of experiment:

Table 2.172 Factor variation intervals

Factors	Variation levels			Variation intervals Δx
	–	0	+	
x_1-temperature	142	147	152	5
x_2-concentration C	35	37.5	40	2.5
x_3-reaction time	7	8.5	10	1.5

The *FUFE* design with outcomes is given in Table 2.173.

Table 2.173 FUFE 2^3

No.	Design matrix			Response yield C % y_u
	X_1	X_2	X_3	
1	–	–	–	55.9
2	–	–	+	63.3
3	–	+	–	67.5
4	–	+	+	68.8
5	+	–	–	70.6
6	+	–	+	68.0
7	+	+	–	68.6
8	+	+	+	62.4

By applying formulas (2.62) to (2.64) we get:

b_0=65.64; b_1=1.76; b_2=1.19; b_3=-0.01;
b_{12}=-3.09; b_{13}=-2.19 b_{23}=-1.21 and b_{123}=0.31.

It is evident that the obtained effects of three even interactions are large when compared to linear effects. This means that the response surface is curved and that the optimum is near. To describe the optimum by a second-order model it is necessary, in accord with *CCOD*, to upgrade the basic *FUFE* 2^3 to *CCOD*. Seven additional design points are needed for this: Six in starlike points and one in the design center. Variation levels for additional design points are given in Table 2.174.

Table 2.174 Factor-variation intervals

Factors	Variation levels		
	-2	0	+2
x_1-temperature	137	147	157
x_2-concentration C	32.5	37.5	42.5
x_3-reaction time	5.5	8.5	11.5

The design matrix for additional design points with experimental outcomes is given in Table 2.175.

Table 2.175 Additional design points for CCOD

No.	Design matrix			Response yield C %
	X_1	X_2	X_3	y_u
9	0	0	0	66.9
10	2	0	0	65.4
11	-2	0	0	56.9
12	0	2	0	67.5
13	0	-2	0	65.0
14	0	0	2	68.9
15	0	0	-2	60.3

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Six design points in the experimental center had to be done due to Table 2.164. For the sake of economy only one design point in the experimental center was set in this example, since the reproducibility variance was obtained in the basic experiment. By processing all 15 design points, the following regression coefficients for second-order model were obtained:

b_0=67.711; b_1=1.944; b_2=0.906; b_3=1.069; B_{11}=-1.539;
b_{22}=-0.264; b_{33}=-0.676; b_{12}=-3.088; b_{13}=-2.188; B_{23}=-1.212.

The second-order regression model has this form:

$$\widehat{y} = 67.711 + 1.944X_1 + 0.906X_2 + 1.069X_3 - 1.539X_1^2 - 0.264X_2^2 - 0.676X_3^2 - 3.088X_1X_2 - 2.188X_1X_3 - 1.212X_2X_3 \quad (2.120)$$

2.3.4 D-optimality, B_k-designs and Hartley's Second-order Designs

D-optimality designs are very attractive for researchers, as their application provides maximal accuracy in estimating regression coefficients. The price for higher accuracy is paid by an increased number of design points, which explains why such designs are less frequently used in practice. The *continuous D-optimality design* [34], constructed on a k-dimension cube, has this formula for the number of design points [34]:

$$N = 2^k + k \times 2^{k-1} + \frac{k(k-1)}{2} \times 2^{k-2} \tag{2.121}$$

where:
k-is the number of factors.

In the given case, experimental points are in hypercube crowns, in the middle of the edges and in the center of two-dimensional planes.

Kono's designs [46] are attempts to reduce the number of design points in continuous D-optimality designs (constructed on a hypercube), by replacing all points in centers of two-dimensional planes with one point in the hypercube center. The number of design points by Kono's designs is defined by this expression:

$$N = 2^k + k \times 2^{k-1} + 1 \tag{2.122}$$

Tables with practical formulas for calculating regression coefficients in Kono's designs are known [46]. The design matrix and coefficients for calculating regression coefficients for Kono's k=2 and k=3 designs are given in Tables 2.176 and 2.177. Analogous data for k=4 are given in reference [46]. This reference contains a dispersion matrix that may serve to check the statistical significance of regression coefficients for Kono's designs with different numbers of factors. Let us analyze an actual application of Kono's design for obtaining a second-order regression model for k=2. This experiment includes nine design points, with the design shown in Table 2.176.

Table 2.176 Kono's design $2^2 + 2x2^{2-1} + 1$

No. trials	X_1	X_2	Coefficients						$\bar{y}_u$	$\hat{y}_u$	$\left(\bar{y}_u - \hat{y}_u\right)^2$
			b_0	b_{11}	b_{22}	b_1	b_2	b_{12}			
1	0	0	0.5772	-0.3234	-0.3234	0	0	0	62	62.4	5.76
2	+	+	-0.1057	0.1691	0.1691	0.1961	0.1961	0.25	50	48.5	2.25
3	−	+	-0.1057	0.1691	0.1691	-0.1961	0.1961	-0.25	67	67.86	0.64
4	−	−	-0.1057	0.1691	0.1691	-0.1961	-0.1961	0.25	60	62.2	4.84
5	+	−	-0.1057	0.1691	0.1691	0.1961	-0.1961	-0.25	70	70.0	0
6	+	0	0.2114	0.1617	-0.3383	0.1078	0	0	56	58.6	6.76
7	0	+	0.2114	-0.3383	0.1617	0	0.1078	0	60	61.1	1.21
8	−	0	0.2114	0.1617	-0.3383	-0.1078	0	0	70	64.4	31.36
9	0	−	0.2114	-0.3383	0.1617	0	-0.1078	0	73	71.8	1.44
Sum											54.26

Kono's design matrix corresponds to second-order orthogonal design matrices so that in this case experimental outcomes from Example 2.51, Table 2.165, were used. The value of regression coefficient b_0 is determined by adding coefficient yields of the associated column to the response column:

$$b_0=0.5772\times62-0.1057\times50-0.1057\times67-0.1057\times60-0.1057\times70+0.2114\times56 +0.2114\times60+0.2114\times70+0.2114\times73=64.43$$

Values of other regression coefficients are determined in the same way, so that the regression model becomes:

$$\widehat{y}=64.43-2.88X_1-3.95X_2-6.80X_1X_2-2.90X_1^2+0.60X_2^2 \quad (2.123)$$

To check lack of fit of the regression model, its variance is determined:

$$S_{AD}^2=\frac{\sum_1^9 25\left(\bar{y}_u-\widehat{y}_u\right)^2}{9-6}=\frac{25\times54.26}{3}=452.17$$

Where the reproducibility variance is S_y^2=375.0, the arithmetic value of *Fisher's* criterion is: F_R=1.21.

Table 2.177 Kono's design $2^3+3x2^{3-1}+1$

No. trials	X_1	X_2	X_3	Coefficients									
				b_0	b_{11}	b_{22}	b_{33}	b_1	b_2	b_3	b_{12}	b_{13}	b_{23}
1	0	0	0	0.6554	-0.2479	-0.2479	-0.2479	0	0	0	0	0	0
2	+	+	+	-0.0861	0.0630	0.0630	0.0630	0.0804	0.0804	0.0804	0.0979	0.0979	0.0979
3	+	−	+	-0.0861	0.0630	0.0630	0.0630	0.0804	-0.0804	0.0804	-0.0979	0.0979	-0.0979
4	−	−	+	-0.0861	0.0630	0.0630	0.0630	-0.0804	-0.0804	0.0804	0.0979	-0.0979	-0.0979
5	−	+	+	-0.0861	0.0630	0.0630	0.0630	-0.0804	0.0804	0.0804	-0.0979	-0.0979	0.0979
6	+	+	−	-0.0861	0.0630	0.0630	0.0630	0.0804	0.0804	-0.0804	0.0979	-0.0979	-0.0979
7	+	−	−	-0.0861	0.0630	0.0630	0.0630	0.0804	-0.0804	-0.0804	-0.0979	-0.0979	0.0979
8	−	−	−	-0.0861	0.0630	0.0630	0.0630	-0.0804	-0.0804	-0.0804	0.0979	0.0979	0.0979
9	−	+	−	-0.0861	0.0630	0.0630	0.0630	-0.0804	0.0804	-0.0804	-0.0979	0.0979	-0.0979
10	+	0	+	0.0861	0.0620	-0.1880	0.0620	0.0446	0	0.0446	0	0.0542	0
11	0	−	+	0.0861	-0.1880	0.0620	0.0620	0	-0.0446	0.0446	0	0	-0.0542
12	−	0	+	0.0861	0.0620	-01880	0.0620	-0.0446	0	0.0446	0	-0.0542	0
13	+	−	0	0.0861	0.0620	0.0620	-0.1880	0.0446	-0.0446	0	0.0542	0	0
14	−	−	0	0.0861	0.0620	0.0620	-0.1880	-0.0446	-0.0446	0	0.0542	0	0
15	−	+	0	0.0861	0.0620	0.0620	-0.1880	-0.0446	0.0446	0	-0.0542	0	0
16	+	+	0	0.0861	0.0620	0.0620	-0.1880	0.0446	0.0446	0	0.0542	0	0
17	0	+	+	0.0861	-0.1880	0.0620	0.0620	0	0.0446	0.0446	0	0	0.0542
18	+	0	−	0.0961	0.0620	-0.1880	0.0620	0.0446	0	-0.0446	0	-0.0542	0
19	0	−	−	0.0861	-0.1880	0.0620	0.0620	0	-0.0446	-0.0446	0	0	0.0542
20	−	0	−	0.0861	0.0620	-0.1880	0.0620	-0.0446	0	-0.0446	0	0.0542	0
21	0	+	−	0.0861	-0.1880	0.0620	0.0620	0	0.0446	-0.0446	0	0	-0.0542

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In practice, we often use designs that are very similar to D-optimality designs in their properties, but that contain a smaller number of design points. Such designs are known as B_k and *Hartley's* designs.

B_k-designs are constructed on a k-dimension cube with an equal number of design points in cube crowns and in the center (k+1) of edges [34].

From the practical point of view, design B_4 is also interesting as it includes only 24 design points, i.e. 2^4 $FUFE$ design points (points in hypercube crowns) and additional 8 design points in starlike points. The matrix of such a design is shown in Table 2.178.

This regression model is obtained by processing experimental outcomes [47]:

$$\begin{aligned}\widehat{y} = {} & 4.724 + 0.177X_1 + 0.327X_2 - 0.155X_3 + 0.151X_4 \\ & -0.577X_1^2 - 0.114X_2^2 + 0.033X_3^2 - 0.244X_4^2 + 0.211X_1X_2 \\ & -0.119X_1X_3 + 0.133X_1X_4 + 0.142X_2X_3 + 0.261X_2X_4 + 0.223X_3X_4\end{aligned} \tag{2.124}$$

Table 2.178 B_4 design

No. trials	X_1	X_2	X_3	X_4	$\bar{y}_u$
1	−	−	−	−	12.4
2	−	−	−	+	20.7
3	−	−	+	−	13.1
4	−	−	+	+	27.5
5	−	+	−	−	45.1
6	−	+	−	+	60.6
7	−	+	+	−	44.8
8	−	+	+	+	58.9
9	+	−	−	−	15.0
10	+	−	−	+	19.5
11	+	−	+	−	10.8
12	+	−	+	+	20.2
13	+	+	−	−	39.2
14	+	+	−	+	56.7
15	+	+	−	−	41.4
16	+	+	+	+	63.5
17	+	0	0	0	31.3
18	−	0	0	0	34.9
19	0	+	0	0	49.9
20	0	−	0	0	26.3
21	0	0	+	0	37.9
22	0	0	−	0	41.3
23	0	0	0	+	44.6
24	0	0	0	−	30.8

Table 2.179 H_{A5}-design

No. trials	X_1	X_2	X_3	X_4	X_5
1	+	+	+	+	+
2	−	+	+	+	−
3	+	−	+	+	−
4	−	−	+	+	+
5	+	+	−	+	−
6	−	+	−	+	+
7	+	−	−	+	+
8	−	−	−	+	−
9	+	+	+	−	−
10	−	+	+	−	+
11	+	−	+	−	+
12	−	−	+	−	−
13	+	+	−	−	+
14	−	+	−	−	−
15	+	−	−	−	−
16	−	−	−	−	+
17	+	0	0	0	0
18	−	0	0	0	0
19	0	+	0	0	0
20	0	−	0	0	0
21	0	0	+	0	0
22	0	0	−	0	0
23	0	0	0	+	0
24	0	0	0	−	0
25	0	0	0	0	+
26	0	0	0	0	−
27	0	0	0	0	0

Hartley's designs include design points in cube crowns (regular fractional replica), as well as "*starlike*" and null design points. Design H_{A5} (k=5) is of particular importance for being especially efficient. The total number of design points for such a design is not great and it is N=27. Its matrix is given in Table 2.179.

2.3.5 Conclusion after Obtaining Second-order Model

After obtaining an optimal mathematical model of a research subject, a conclusion is brought down to a statement on lack of fit or inadequacy of the obtained second-order regression model. The significance of regression coefficients in second-order models is marginal in comparison with their significance in linear models, since purposes of obtaining linear and nonlinear models differ.

As said before, linear models are used to reach (move towards) optimum, so that the significance of regression coefficients is an assumption for successful application of the steepest ascent method. Linear models, therefore, include as many factors as possible, and full factorial experiments are even replicated with increased factor variation intervals.

With nonlinear models, which are aimed at mathematical modeling or adequate description of the optimum region and that as a rule have numerous regression coefficients, rejection of insignificant regression coefficients is not so important as in the phase of linear modeling. For second-order models, an estimate of lack of fit or inadequacy of the model is of particular importance.

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Inadequate second-order model

As with an inadequate linear model, it is possible to switch to a higher order or a third-order model. Realization, processing of experimental results and analysis and interpretation are very complicated for third-order designs, which makes such a suggestion not efficient enough [16].

Removing the model inadequacy by introducing rejected factors (in the phase of screening experiments and linear model analysis) and by an increased number of trial replications, is much more acceptable.

Adequate second-oder model

When processing of experimental outcomes shows an adequate regression model, the problem of mathematical modeling of response optimum is terminated, since an interpolation model of the research subject has been obtained.

In the case of an extreme experiment, we are faced with determining optimum coordinates from the obtained mathematical model. In that case, canonical analysis or methods of nonlinear programming are mostly used. The obtained optimum coordinates on a research, lab level are the starting point for a switch from lab to pilot-plant or full-scale levels[3)]. The procedure is in principle repeated in a larger sys-

3) "Scale-Up" is an Anglo-Saxon term for switch from smaller to lager-scale systems

tem within the defined experimental center /48, 18/. The obtained mathematical model and coordinates of the optimum are particularly useful for problems of process control in a full-scale plant.

2.4 Statistical Analysis

2.4.1 Determination of Experimental Error

A researcher who manages an experimental study by application of design of experiments, must have a clear idea on methods of processing experimental results in the actual case and before starting it, so as to facilitate defining the research objective and the drawing of conclusions. A mathematical theory of experiments differentiates several types of *errors* in processing experimental results, each of which is characteristic of a definite phase of data analysis. Each experiment consists of a certain number of *design points-trials*, each design point-trial of one or more *replicated trials*, and a single design point of one or more *replicated measurements (determinations)*. In accordance with this we distinguish *experiment error (reproducibility variance)*, *trial error-variance of replicated trials* and *measurement error (determination error)*. Measurement error is local to a trial, and may come either from an instrument or from specimen differences (sampling error) or both. It can be reduced by taking repeated measurements and averaging. Whether or not such averaging is worthwhile, depends on its magnitude with respect to the trial error. The standard deviation of measurement error may be estimated from repeated measurements within the same trial. The measurement variance is the square of the standard deviation of the measurement error. Recognition of the design point-trial and experimental errors is necessary for a verification of the significance of regression coefficients and lack of fit of a regression model.

Measurement-determination error has been discussed in Sect. 2.1.4.

Trial error and experimental error also belong to the group of *random errors* so that in estimating their values we use the same approach as for random measurement errors. In determining a measurement error we take into account the number of replicate measurements (u); in a trial error the number of replicate trials (n), and in an experimental error the number of different trials (N). Replication of a trial must not be mixed up with replication of measurements in one trial. When determining a trial error, we estimate the standard deviation of replicated trials. This may be estimated by calculating the standard deviation of several trials whose control factor settings are the same. Ideally one would set up and run the same trial repeatedly. The small differences in a setup are an important component of the replicate error. The replicate error is made up of two parts: trial error and measurement error. The replicate variance is just the square of the replicate standard deviation. In the case of experimental error we estimate the variance of reproducibility. Prior to calculating experimental error it is necessary to check variance homogeneity of different trials

III
Mixture Design "Composition-Property"

3.1
Screening Design "Composition-Property"

To begin any kind of experimental study one should review the reference literature and discuss problems with researchers from the field. A result of the mentioned preliminary study of a research subject is mostly a long list of potential factors of a system. Modern strategy of experimental research, as has been described in previous chapters, begins with screening experiments, their task being to find the most significant factors for the observed system. Screening experiments most frequently solve the lowest level of a research objective i.e. screening out factors by the significance of their effects on a response system. More complex research objectives require a mathematical model of the researched phenomenon, which is brought down to constructing a regression model and to an analysis of the obtained response surface. Screening experiments offer sufficient data for obtaining a linear regression model.

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$$E(y)=\beta_0+\beta_1X_1+\beta_2X_2+...+\beta_pX_p \tag{3.1}$$

Relative values of factor effects are proportional to the values of regression coefficients β_I, of the associated coded factors X_i. A factor may be statistically insignificant if the value of the associated regression coefficient is small enough. Part of the philosophy of screening experiments from sect. 2.2 is transferred to selecting components of a mixture or composition. The main difference lies both in the concept of screening and in construction of a design of experiments matrix. The difference exists in specific constraints on the component ratio in the mixture. In *mixture* experiments, response (y) is a function of the q-components ratio only (X_i) and not of its total quantity. Constraints refer to the ratios of each q-component and to the sum of ratios of all components. It is mathematically expressed in this way:

$$0 \le X_i \le 1.0; \sum_{i=1}^{q} X_i = 1.0 \tag{3.2}$$

It is evident that components levels-ratios are not independent and that the level of one component depends on levels of q-1 components. Screening experiments are recommended in situations when the number of components is q>6. For five or

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fewer components, fitting it with a second-order model is acceptable [1]. A design matrix for a five-component system and a second-order model usually contains 20 to 25 trials-design points, which is acceptable. For a second-order model for six or more mixture components, the number of design points-trials of a design matrix is large, especially if one accounts for the fact that it is uncertain whether all mixture components are significant and whether all the selected variation levels of component ratios are important. Due to the complexity, a screening experiment with a small number of trials is necessary. Screening out of components in the mixture is brought down to an analysis of the response surface or to response in all directions of the factor space. A response-surface analysis or response-value analysis means determining directions, or axes of the factor space where response values are constant, close to being constant or change only a little. It is an evident assumption that significant mixture components have great linear effects. This is correct in principle, but there are exceptions. It is therefore suggested [2] to include into the screening design of experiments at least one trial in its center in order to catch nonlinearity, which may be present. Linear regression models by *Scheffe* [3] have the form:

$$E(y)=\beta_1X_1+\beta_2X_2(+...+\beta_pX_p \tag{3.3}$$

Constant β_0 is omitted due to the limitation that the sum of ratios of all components is equal to one. Screening out of components is brought down to finding components Xi that have *no effects* and/or have *the same effects* on the property-response of the mixture:

- If the value of coefficient β_i is equal to the mean of coefficients in the model, then E_i=0;

$$E_i = \beta_i - (q-1)^{-1}\sum_{i\neq j}\beta_j \tag{3.4}$$

- or y values do not vary in any direction normal to X_i=0 (simplex basis). In other words, there is no change of y in the direction of the normal passing through the simplex centroid. Geometrically, this is a one-dimensional local space where X_j=(1-X_i)/(q-1) for each j≠i. Value E_i is all over the simplex called linear factor effect X_i. Constant responses in a direction parallel to the X_i-axis (in the direction of X_i) indicate the insignificance of the factor-component X_i.
- When two or more coefficients are equal (e.g. β_1=β_2=β_6) then the effects of associated components are equal in the experimental region. The associated sum may be considered as one component, which means a reduction in the number of basic components. This also means that there is no variation of y in the q-dimensional local space of simplex, where "e" is the number of components with equal effects.

Example 3.1

Examples of response surfaces for the two mentioned cases are shown in Figs. 3.1 and 3.2.

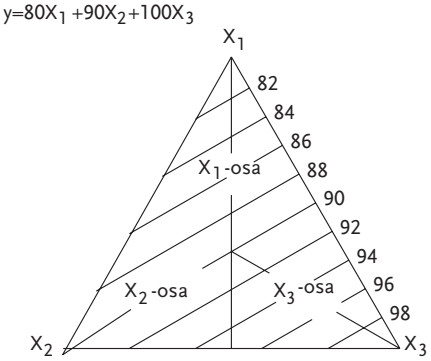

Figure 3.1 Response surface of a three-component

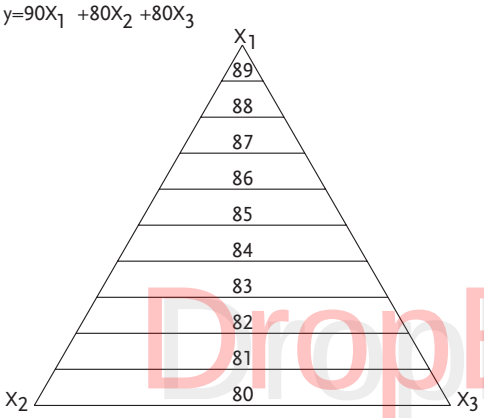

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Figure 3.2 Response surface of a three-component

The response surface in Fig, 3.1 is defined by the regression model $\widehat{y} = 80X_1 + 90X_2 + 100X_3$, where we can see that there is no variation of y in direction X_2:

β_2-(β_1+β_3)/2=90-(80+100)/2=0

Contour or isoresponse lines are parallel to the X_2-axis. The response surface in Fig. 3.2 is defined by the regression model $\widehat{y} = 90X_1 + 80X_2 + 80X_3$. It is evident that the regression coefficients for components X_2 and X_3 are equal (β_2=β_3) and that there are no response changes in the directions of response isolines for X_2+X_3=const.

Example 3.2

Regression coefficients in the regression model $\widehat{y} = 80X_1 + 90X_2 + 90X_3 + 100X_4$ are:

β_2=β_3; β_2=(β_1+β_3+β_4)/3; β_3=(β_1+β_2+β_4)/3

Preliminary analysis shows that in this case the mixture has components that have the same but statistically insignificant effects. Effects of components X_1 and X_4, are significant, while effects of components X_2 and X_3 are equal and insignificant. In Example 3.5, where a mixture of eight components is analyzed, there exist separately components with no effects and components with equal effects. The effect of component X_i may be analyzed more generally than the definition given by Eq. (3.4). Let us observe a matrix with components that have limitations in variation: $0 \le a_i \le X_i \le b_i \le 1$ Variation effects of components in such situations are defined by this expression:

$$E_i = R_i \left[\beta_i - (q-1)^{-1} \sum_{i \neq j} \beta_j \right] \tag{3.5}$$

where:
$R_i = b_i - a_i$.

Cox [3] has in his work developed a different expression for the effect of component X_i in a linear model:

$$E_i = R_i \left(\frac{1}{1-s_i} \right) \left(\beta_i - \sum_{i=j}^{q} \beta_j s_j \right) \tag{3.6}$$

It should be noted that *Cox*'s approach explicitly defines the *standard-referential* mixture or standard referential composition.

Hence, in expression (3.6): $s=(s_1, s_2, \ldots, s_q)$ is the referential composition. If referential composition is in the simplex centroid, then $s_1=\ldots=s_q=1/q$, so that expression (3.6) is transformed into expression (3.5). Actually, all referential compositions lying on an axis of the i-th component (except those in the vertex where $s_i=1$) will have E_i effects as defined by expression (3.5) and will be measured in the direction parallel to the axis of the i-th component, or normal to the simplex basis opposite of the i-th vertex. That is, the effect of component X_i is measured normally to the local space of the remaining components. It should be pointed out that in the case of variations of component ratios with no limitations, the effect of component X_i is determined parallel to the X_i-axis, orthogonally for the local factor space of the remaining components. The simplex centroid has a unique meaning as it is the point that lies on all the axes or components. It should also be noted that directions in which effects are determined do not depend on component transformations into pseudo-components, or vice versa. Referential compositions outside the simplex centroid have E_i effects that are determined in the direction of the line passing through the vertex of pure X_i but not parallel to the i-th component or axis. Such a direction, for example, suits the determining of the effect for component X_i that has been physically added to the existing referential composition, which has fixed quantities of other components. In Figs. 3.1 and 3.2 coefficient contrasts are absolutely equal to zero, so that there is no doubt that the response is constant in the associated directions. This is not the case in practice, so that a statistical procedure is defined to establish which of the contrasts is statistically significantly different from zero. Contrasts in the matrix form may be marked in this way $C\beta$, so that the variance of linear contrast of regression coefficients is:

$$V(C\beta) = \sigma^2 C(X'X)^{-1}C' \tag{3.7}$$

where:
σ^2-is error variance;
C-is coefficient vector that defines the contrast;
X-is matrix (n×q) of component ratios;
$\sigma^2(XX)^{-1}$-is the variance of the covariance matrix of regression coefficients.

Example 3.3 [4]
Vector C for contrast β_2-(β_1+β_3)/2 has this form: C=(-0.5; 1; -0.5). Null hypothesis H_0: Cβ=0 is tested by Students t-test:

$$t = C\beta / \left[\sigma^2 C(X'X)^{-1}C'\right]^{0.5} \tag{3.8}$$

where degree of freedom corresponds to variance σ^2.

For each interesting contrast, one can do the given test, and regression model can be reduced in accord with its results. F-test for a difference between residual mean sum of squares of a full and reduced regression model is also possible. Error variance or reproducibility variance may also be determined as before, from replicate design points or from previous experiments. It should be stressed that the analyzed statistical testing is secondary in screening experiments of the "mixture" type, and that the primary thing is the ranking of effects or component regression coefficients.

3.1.1
Simplex Lattice Screening Designs

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The efficiency of screening experiment designs depends on the form of experimental domain. If this domain suits a *total simplex* (0≤X≤1; i=1, 2,..., q), then a design of experiments with (2q+1) design points-trials is recommended. In that case design of experiments includes q-pure components (X_i=1.0), of centroid simplex ($X_i = q^{-1}$ for all i=1, 2,..., q) and q-internal points with coordinates: $\left[(2q)^{-1}, (2q)^{-1}, \ldots, (q+1)/2q, \ldots, (2q)^{-1}\right]$.

It should be said that q-responses of pure components makes determination of regression coefficients of linear model possible, while q-internal and central points serve to estimate the nonlinearity of the response surface. It is useful to include in the mentioned designs of experiments q-points of *"null effects"* in this form:

$$\left[(q-1)^{-1}, (q-1)^{-1}, \ldots, 0, \ldots, (q-1)^{-1}\right]$$

Null effects are included in situations when it is expected that absence of a component may have a strong effect on the response level. Designs of experiments, which include null effects have (3q+1) design points-trials. Such designs belong to *simplex lattice screening designs* and should not be mixed up with *scheffe simplex lattice designs* [5] and *simplex centroid designs* [6], which have considerably more trials (for q>5) and facilitate the fitting of more complicated models. As is known, simplex screening designs contain four groups of trials, here given in Table 3.1.

Mark	Name	Number	Composition
A	Vertex	**q**	$X_i = 1, X_j = 0;$ each $j \neq i$
B	Internal	**q**	$X_i = (q+1)/2q; X_j = (2q)^{-1};$ each $j \neq i$
C	Centroid	**1**	$X_i = q^{-1};$ each i
D	null effects	**q**	$X_i = 0; X_j = (q-1)^{-1};$ each $j \neq i$

Table 3.1 Simplex screening designs

Name		Design matrix						
Code	**Group**	**No. trials**	X_1	X_2	.	.	.	X_q
A	**Vertex**	1	1	0	.	.	.	0
		2	0	1	.	.	.	0
		.	.	.	.	.	.	.
		.	.	.	.	.	.	.
		.	.	.	.	.	.	.
		Q	0	0	.	.	.	1
B	**Internal**	1	(q+1)/2q	1/2q	.	.	.	1/2q
		2	1/2q	(q+1)/2q	.	.	.	1/2q
		.	.	.	.	.	.	.
		.	.	.	.	.	.	.
		.	.	.	.	.	.	.
		Q	1/2q	1/2q	.	.	.	(q+1)/2q
C	**Centroid**	1	q^{-1}	q^{-1}	.	.	.	q^{-1}
D	**Null effects**	1	0	$(q-1)^{-1}$	.	.	.	$(q-1)^{-1}$
		2	$(q-1)^{-1}$	0	.	.	.	$(q-1)^{-1}$
		.	.	.	.	.	.	.
		.	.	.	.	.	.	.
		.	.	.	.	.	.	.
		Q	$(q-1)^{-1}$	$(q-1)^{-1}$	.	.	.	0

Graphic analysis of simplex screening experiments

Graphic analysis of simplex screening designs is brought down to the graphic presentation of response in the direction of each axis or component. When a design of experiments has all four groups of trials, then on each axis there are four compositions-mixtures. An example of graphic analysis of a three-component composition of a rocket propellant is given in the work of *Kurotori* [7] in Fig. 3.3.

The elasticity of a rocket propellant was measured as the system response. An analysis of Fig. 3.3 indicates that all three components of the composition have large effects and that the response surface is greatly curved or nonlinear. Besides, the greatest response values were obtained close to the centroid. Large response values also occurred for lower X_1 levels and for X_3 values between 0.33 and 0.67. Graphic interpretation of the simplex screening experiment may be compared to the response surface contour plots of the same example, Fig. 3.4.

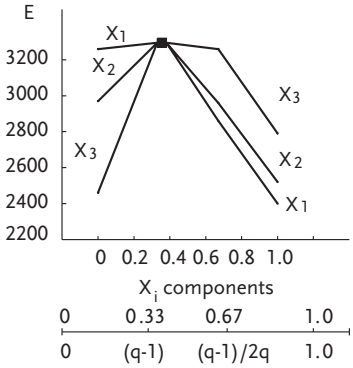

Figure 3.3 Three-component mixture response

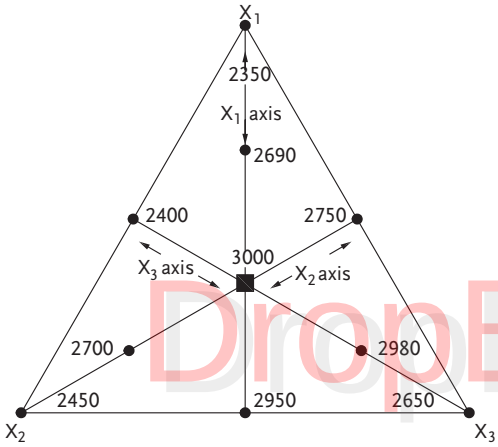

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Figure 3.4 Response for a three-component composition of rocket propellant

It is evident that graphic interpretation of the simplex screening experiment offers a robust idea about the form of the response surface. It is therefore not recommended to replace the response surface contour plot with a graphic interpretation of the simplex screening experiment.

Example 3.4

A simplex screening experiment was done for selection of a ten-component composition of petrol. The outcomes of the experiment are given in Table 3.2. All four groups of design points have been included in this ten-component design of experiments. The design included 3×10+1 trials or 31 mixtures. Values of linear regression coefficients and of effects are given in Table 3.2. Graphic interpretation of this ten-component simplex screening experiment is shown in Fig. 3.5.

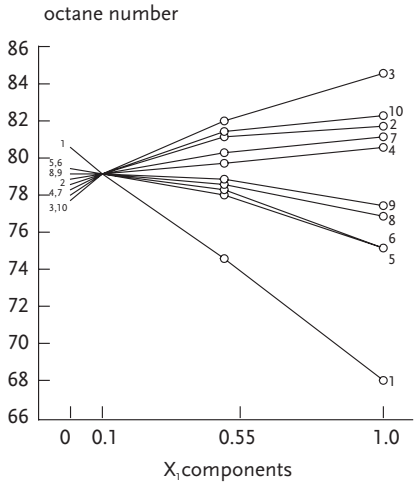

Figure 3.5 Simplex screening diagram for a ten-component mixture

Table 3.2 Simplex screening experiment q=10

Components		Composition			Regression coefficients	
No.	**Code**	**Null effects**	**Internal**	**Vertex**	β_i	E_i (Eq. 3.4)
1	LSR	80.9	74.3	66.6	67.5	-13.1
2	REFA	79.4	81.4	81.8	82.5	3.6
3	REFB	79.0	82.2	84.5	85.0	6.4
4	LLCC	79.3	80.8	80.9	81.5	2.5
5	LHCCA	79.7	78.3	76.2	76.8	-2.7
6	LHCCB	79.7	78.5	76.2	76.9	-2.6
7	HLCC	79.3	80.9	81.3	81.9	2.9
8	HHCCA	79.6	78.6	78.1	78.4	-0.9
9	HHCCB	79.6	78.8	78.3	78.7	-0.7
10	POLY	78.7	82.0	82.5	83.3	4.5
		95% Confidence interval			±1.1	±1.2

Response or property of mixture in centroid is 79.4

Analysis of Fig. 3.5 may bring us to several conclusions:

- Component No. 1, has a great negative effect. Components No. 5, 6, 8, 9 also have negative effects, while components 2, 3, 4, 7 and 10 have positive effects.
- Components 5 and 6, 8 and 9, 4 and 7 and 2 and 10 behave similarly in mixtures. This behavior corresponds to the physical essence of components. There are only minor differences in production of the mentioned components.

- Components 2, 4, 7, 8 and 9 have the lowest effects on the octane number of petrol.
- The response surface of the petrol octane number has small nonlinearity.
- With an increase in number of components q, the centroid is shifted towards the null-effect points $\left[X_i = 0; X_j = (q-1)^{-1}, j \neq i\right]$, and internal points move away from the vertex.

It should be pointed out that to obtain a second-order model we have to do 66 trials, or 10 with pure components, 45 of binary composition, 10 internal points and one centroid point. However, the 21 compositions out of 31 mixtures from a screening experiment are simultaneously a part of the design of 66 design points for a second-order model. This shows that even in mixture experiments we may deal with the principle of upgrading/augmenting a design of experiments.

3.1.2
Extreme Vertices Screening Designs

A large number of "mixture" problems has limitations on component ratios:

$$0 \leq a_i \leq X_i \leq b_i \leq 1;\ i = 1.2,\ldots,q \tag{3.9}$$

Apart from the mentioned limitations, one may even meet a multiple limitation of this form:

$$C_j \leq A_{1j}X_1 + A_{2j}X_2 + \ldots + A_{qj}X_q \leq d_j \tag{3.10}$$

Limitations (3.2); (3.9) and (3.10), geometrically form a *hyperpolyhedron* in factor space. The design of experiments for a linear model, according to *Elfving* [8] actually contains a definite number of hyperpolyhedron vertices. Due to the complicated procedure and criteria, which are used to define vertices-design points, *XVERT* algorithms [9] are nowadays used as software packages for computers. Similar to previous chapter, in order to check nonlinearity of the response surface, a point in the center of experiment or a centroid is included in a design of experiments. The *XVERT* algorithm defines extreme vertices of a design of experiments matrix in this way:

a) Rank the components according to the growth of rank (b_i-a_i):X_1 has the lowest and X_q the highest rank;
b) Define a two-level design of experiments with upper and lower limits of q-1 components with the lowest rank;
c) Calculate the level-ratio of the q-th component:

$$X_q = 1.0 - \sum_{i=1}^{q-1} X_i$$

d) A level is extreme if $a_q \leq X_q \leq b_q$. For vertices outside limitations for X_q take the value of upper or lower limits or the value, which is closer to the calculated value.
e) For each vertex the outside limitations define additional vertices (max.=q-1) with an *adjustment of levels* of associated components X_{q-1} by adding up calcu-

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lated differences of the value X_q and the adopted upper or lower limitation, respectively. Additional vertices are defined only for the components whose adjusted levels remain inside component limitations.

This kind of algorithm defines 2^{q-1} vertices-trials, whereby each vertex generates at most q-1 additional vertices, so that, total possible number of design points is $2^{q-1}+(q-1)2^{q-1}$. A vertex is not generated when levels of just one component are outside limitations. Repeated vertices are dropped in calculations. A repeated vertex may appear only when the last component X_q takes the highest (b_q) or lowest (a_q) level. Vertices of a two-level design of experiments are a subgroup of extreme vertices, obtained from the design 2^{q-1} thus:

a) Calculate X_q levels for all vertices.
b) All vertices for which $a_q \leq X_q \leq b_q$, form the design *core*.
c) Each vertex for which the X_q level is adjusted generates additional vertices that form the *candidate subgroup*.
d) Design of experiment consists of the core and one vertex from each candidate subgroup. In a given subgroup, the vertex that enters the best design of experiments is defined in the way that it represents all the possible combinations of the core vertex and one vertex from each subgroup. If n_i is the number of vertices in the i-th subgroup, then the number of possible designs of experiments is equal to $\prod_{i=1}^{k} n_i$ where k-is number of subgroups.

Example 3.5 [9]
To begin with, we can analyze a three-component composition with these limitations on components:

$0.1 \leq C_1 \leq 0.7; b_1-a_1=0.6; 0.0 \leq C_2 \leq 0.7; b_2-a_2=0.7;\ 0.1 \leq C_3 \leq 0.6;\ b_3-a_3=0.5.$

Ranking or sequence of components due to growth of rank is shown in Table 3.3.

Table 3.3 Components with limitations

Components	Minimum a_i	Maximum b_i	Rank b_i-a_i
$X_1=C_3$	0.1	0.6	0.5
$X_2=C_1$	0.1	0.7	0.6
$X_3=C_2$	0.0	0.7	0.7

Vertices of design core correspond to $2^{q-1} = 2^{3-1} = 2^2$ full factorial design where upper and lower levels correspond to limitations of q-1 factors. Component levels X_q=X_3 are determined thus:

$X_3 = 1.0 - \sum_{i=1}^{q-1} X_i = 1.0 - X_1 - X_2$ Vertices are defined by these ratios of components in Table 3.4.

Vertices B and C have X_3 =0.3 and 0.2, respectively. These values are within limitations 0.0:0.7, for component of X_3. However, X_3 component levels in vertices A and D (X_3=0.8 and -0.3) are outside the limiting interval. Vertices A and D have to be adjusted to satisfy limitations of X_3. The component level of X_3 for vertex A should be reduced by 0.1 to obtain a value of 0.7, which is the upper limit of the value. The component level of X_3 for vertex D has to be increased by 0.3 to obtain the value of 0.0. Components X_1 and X_2 for both vertices have to be corrected to compensate for the changes of X_3. Results of corrections are given in Table 3.5. According to the *XVERT* algorithm we obtained two additional vertices for A and D, which total six extreme vertices, as shown in Fig. 3.6.

Table 3.4 Design of experiments matrix

Vertex	X_1	X_2	X_3
A	0.1	0.1	0.8
B	0.6	0.1	0.3
C	0.1	0.7	0.2
D	0.6	0.7	-0.3

Table 3.5 Corrected levels

Vertices	X_1	X_2	X_3	Correction
A	0.1	0.1	0.8	
A1	0.1	0.2	0.7	+0.1
A2	0.2	0.1	0.7	+0.1
D	0.6	0.7	-0.3	
D1	0.6	0.4	0	-0.3
D2	0.3	0.7	0	-0.3

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Table 3.6 Ratios of components

Vertices	Vertex mark	X_1	X_2	X_3
B	1	0.6	0.1	0.3
C	2	0.1	0.7	0.2
A1	3	0.1	0.2	0.7
A2	4	0.2	0.1	0.7
D1	5	0.6	0.4	0.0
D2	6	0.3	0.7	0.0

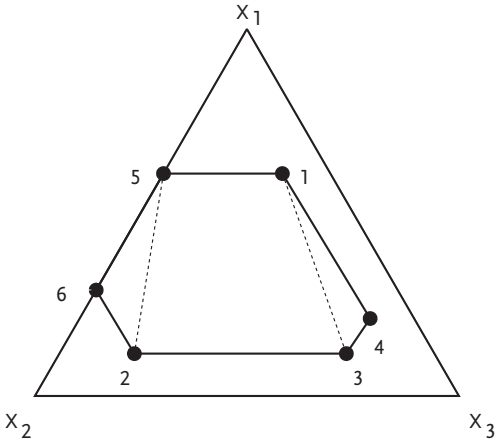

Figure 3.6 Three-component extreme vertices screening experiment

To obtain a design of experiment with four trials-vertices, two candidate subgroups with two vertices each have been generated for A and D vertices, Table 3.6. The design of experiment consists of these vertices 1, 2, (3 or 4) and (5 or 6). Out of four possible vertex combinations, statistically the most efficient is the design of experiment with these vertices-trials 1; 2; 3 and 5.

Example 3.6 [10]

A four-component example is the subject of analysis due to a small number of extreme vertices and the possibility to present all the possible designs of experiments. Limitations for the four mentioned components are as follows:

$0.00 \le X_1 \le 0.04$; $b_1-a_1=0.04$; $0.00 \le X_2 \le 0.10$; $b_2-a_2=0.10$;
$0.40 \le X_3 \le 0.55$; $b_3-a_3=0.15$; $0.40 \le X_4 \le 0.60$; $b_4-a_4=0.20$.

The ratios of components both for 10 extreme vertices and for designs of experiments n=4 and 8 are given in Tables 3.7 and 3.8. Levels or ratios of components X_1, X_2 and X_3 in the design of experiment n=4 are generated from a fractional design 2^{3-1} with generating ratio $X_3=X_1X_2$, Table 3.9.

The calculated level values for X_4 are within limitations for the first three design points. Design point D may be adjusted in two ways (by reducing X_3 for 0.09 and increasing X_4 for 0.09 or by reducing X_2 for 0.09 and increasing X_4 for 0.09), so that two designs of experiments are possible: design with vertices 2, 3, 5 and 8 or design with vertices 2, 3, 5 and 10. Levels or ratios of components X_1, X_2 and X_3 in a design of experiments with 8 trials are generated from *FUFE* 2^3. In this approach two design points have X_4 outside limitations. Each design point may be adjusted in two ways so that $2 \times 2=4$ designs of experiments may be formed.

Table 3.8 shows that designs of experiments generated by *XVERT* software are statistically more efficient than *CADEX* designs.

Table 3.7 Extreme vertices

Vertices	X_1	X_2	X_3	X_4
1	0.00	0.00	0.40	0.60
2	0.00	0.10	0.40	0.50
3	0.04	0.00	0.40	0.56
4	0.04	0.10	0.40	0.46
5	0.00	0.00	0.55	0.45
6	0.04	0.00	0.55	0.41
7	0.00	0.10	0.50	0.40
8	0.04	0.10	0.46	0.40
9	0.00	0.05	0.55	0.40
10	0.04	0.01	0.55	0.40

Table 3.8 Designs of experiment

n	Algorithm	Vertices	G-efficiency
4	All combinations	1,4,6,7	81
	XVERT	2,3,5,8	62
	CADEX	1,2,8,9	28
8	All combinations	1,2,3,4,5,6,7,8	88
	XVERT	1,2,3,4,5,6,7,8	88
	CADEX	1,2,3,5,6,7,8,9	72

CADEX-software for generating designs [10]

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Example 3.7

To do a selection of components in a petrol composition, define the matrix of the extreme vertices screening design. The composition of petrol is made of five components with these:

$0.00 \le X_1 \le 0.10$; $b_1 - a_1 = 0.10$; $0.00 \le X_2 \le 0.10$; $b_2 - a_2 = 0.10$ $0.05 \le X_3 \le 0.15$; $b_3 - a_3 = 0.10$; $0.20 \le X_4 \le 0.40$; $b_4 - a_4 = 0.20$ $0.40 \le X_5 \le 0.60$; $b_5 - a_5 = 0.20$

Table 3.9 Design of experiment n=4

Trials	Vertex	X_1	X_2	X_3	X_4
A	5	0.0	0.0	0.55	0.45
B	3	0.04	0.0	0.40	0.56
C	2	0.0	0.10	0.40	0.50
D		0.04	0.10	0.55	–
D1	8	0.04	0.10	0.46	0.40
D2	10	0.04	0.01	0.55	0.40

Earlier research proved that the linear regression model adequately describes the octane number as a function of component ratio. According to *XVERT* algorithm, design core and candidate subgroups are shown in Table 3.10.

It is interesting to note that the *XVERT* algorithm includes in a design of experiment the first two points from candidate groups. The linear regression model has this form:

$$\widehat{y} = 102.4X_1 + 100.7X_2 + 85.2X_3 + 84.7X_4 + 97.6X_5$$

To use designs that generate the *XVERT* algorithm in practice, consult Table 3.11.

Table 3.10 Design of experiment q=5

Vertices groups	Vertices	X_1	X_2	X_3	X_4	X_5	y
Design core	1	0.10	0.10	0.05	0.20	0.55	95.1
	2	0.10	0.00	0.15	0.20	0.55	93.4
	3	0.00	0.10	0.15	0.20	0.55	93.3
	4	0.10	0.10	0.15	0.20	0.45	94.1
	5	0.00	0.00	0.05	0.40	0.55	91.8
	6	0.10	0.00	0.05	0.40	0.45	91.8
	7	0.00	0.10	0.05	0.40	0.45	92.5
	8	0.00	0.00	0.15	0.40	0.45	90.5
	9	0.00	0.00	0.05	0.35	0.60	92.7
	10	0.10	0.10	0.15	0.25	0.40	93.5
Candidate subgroups							
1	11	0.10	0.00	0.05	0.25	0.60	94.8
	12	0.10	0.00	0.10	0.20	0.60	–
	13	0.10	0.05	0.05	0.20	0.60	–
2	14	0.00	0.10	0.05	0.25	0.60	93.7
	15	0.00	0.10	0.10	0.20	0.60	–
	16	0.05	0.10	0.05	0.20	0.60	–
3	17	0.00	0.00	0.15	0.25	0.60	92.5
	18	0.00	0.05	0.15	0.20	0.60	–
	19	0.05	0.00	0.15	0.20	0.60	–
4	20	0.10	0.10	0.05	0.35	0.40	93.1
	21	0.10	0.05	0.05	0.40	0.40	–
	22	0.05	0.10	0.05	0.40	0.40	–
5	23	0.10	0.00	0.15	0.35	0.40	91.8
	24	0.10	0.00	0.10	0.40	0.40	–
	25	0.05	0.00	0.15	0.40	0.40	–
6	26	0.00	0.10	0.15	0.35	0.40	91.6
	27	0.00	0.10	0.10	0.40	0.40	–
	28	0.00	0.05	0.15	0.40	0.40	–

Table 3.11 Design of experiments by XVERT algorithm

Mark	q	N	n	G-efficiency
A	4	10	4	62
			8	84
B	5	20	8	84
			11	77
			16	88
C	5	28	8	83
			12	71
			12	63
			16	95
D	6	49	8	78
			12	79
			16	82
			16	79
			20	77
E	6	60	7	84
			7	84
			11	79
			19	78
			19	78
F	6	36	9	59
			9	60
			11	51
			11	52
			14	77
			14	64
G	7	109	8	76
			12	70
			12	77
			16	69
			16	71

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Example 3.8

In developing a new eight-component, product criteria of the economy demand that the composition should include these four components: X_1, X_2, X_5 and X_6. The effects of the other four components are unknown. Limitations of components are these:

$0.10<X_1<0.45$; $0.05<X_2<0.50$; $0.00<X_3<0.10$; $0<X_4<0.10$;
$0.10<X_5<0.60$; $0.05<X_6<0.20$; $0.00<X_7<0.05$; $0<X_8<0.05$.

The design of the experiment with outcomes is given in Table 3.12.

Table 3.12 Extreme vertices screening design

Trials	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	y
1	0.100	0.50	0.00	0.00	0.10	0.20	0.05	0.05	30
2	0.100	0.05	0.00	0.00	0.55	0.20	0.05	0.05	113
3	0.100	0.50	0.00	0.10	0.10	0.20	0.00	0.00	17
4	0.150	0.05	0.00	0.10	0.60	0.05	0.05	0.00	94
5	0.100	0.05	0.10	0.00	0.55	0.20	0.00	0.00	89
6	0.100	0.50	0.10	0.10	0.10	0.05	0.00	0.05	18
7	0.100	0.05	0.10	0.10	0.55	0.05	0.00	0.05	90
8	0.400	0.05	0.10	0.10	0.10	0.20	0.05	0.00	20
9	0.350	0.05	0.10	0.10	0.10	0.20	0.05	0.05	21
10	0.300	0.50	0.00	0.00	0.10	0.05	0.00	0.05	15
11	0.100	0.50	0.10	0.00	0.20	0.05	0.05	0.00	28
12	0.450	0.05	0.00	0.00	0.45	0.05	0.00	0.00	48
13	0.450	0.20	0.00	0.10	0.10	0.05	0.05	0.05	18
14	0.450	0.15	0.00	0.10	0.10	0.20	0.00	0.00	7
15	0.450	0.25	0.10	0.00	0.10	0.05	0.05	0.00	16
16	0.450	0.10	0.10	0.00	0.10	0.20	0.00	0.05	19
17	0.259	0.222	0.05	0.05	0.244	0.125	0.025	0.025	38
18	0.259	0.222	0.05	0.05	0.244	0.125	0.025	0.025	30
19	0.259	0.222	0.05	0.05	0.244	0.125	0.025	0.025	35
20	0.259	0.222	0.05	0.05	0.244	0.125	0.025	0.025	40

* chosen conditions for central points 17–20 are average levels of the previous 16 design points

Regression coefficients of linear model have these values:

β_1=-33.3; β_2=-10.3; β_3=-2.7; β_4=-19.7; β_5=150.4; β_6=-46.6; β_7=165.5; β_8=188.6.

It is evident that the effects of components X_1, X_2, X_3, X_4 and X_6 are negative, and those of components X_5, X_7 and X_8 positive. By their size, the effects of components have this sequence: X_5, X_1, X_2, X_4, X_8. Components X_3, X_6 and X_7 have small effects. It has been said that for reasons of economy, the composition of the product should include components X_1, X_2, X_5 and X_6. Since by the definition of a research objective, one should increase response y, it is necessary to exclude components X_3 and X_4 from the composition and keep components X_1, X_2 on lower levels. An analysis of regression coefficients shows that one can establish these approximate relations:

$\beta_1=\beta_4$; $\beta_2=\beta_3$; $\beta_5=\beta_7=\beta_8$

Such approximate relations suggest modeling the system as a three-component mixture.

$Z_1=(X_1+X_4)/(1-X_6)$;
$Z_2=(X_2+X_3)/(1-X_6)$;
$Z_3=(X_5+X_7+X_8)/(1-X_{6)}$.

3.2
Simplex Lattice Design

When studying the properties of a q-component mixture, which are dependent on the component ratio only, the factor space is a regular, (q-1) simplex, and for the mixture the relationship holds

$$\sum_{i=1}^{q} X_i = 1; 0 \leq X_i \leq 1 \tag{3.10a}$$

where
X_i-is the component concentration-ratio-productions, and
q-is the number of components in the mixture.

For binary systems, (q=2) the simplex of dimension 1 is a straight line-segment. For q=3, the regular 2-simplex is an equilateral triangle with its interior. Each point in the triangle corresponds to a certain composition of the ternary system, and conversely each composition is presented by one distinct point. The composition may be expressed as a molar, weight, or volume fraction or a percentage. Vertices of the triangle represent pure substances, and the sides represent binary mixtures. If we draw an altitude from each vertex, then dissect each altitude into ten equal segments and draw through the division points straight lines parallel to the triangle sides, we shall have a triangular network-*simplex lattice*. Approaching from a side to the opposite vertex corresponds to the proportional increasing in content of the "vertex" component, therefore the sequential transfer from one parallel line of two components to another signifies the growth of a third component by 10 per cent, Fig. 3.7. In actual practice though, no altitudes are drawn, instead the component contents are marked off directly on the triangle sides. Such a method of counting is adopted in *Gibbs's* triangle. In *Rozebum's* triangle, the composition of a ternary system is read from three segments of one, Fig. 3.8.

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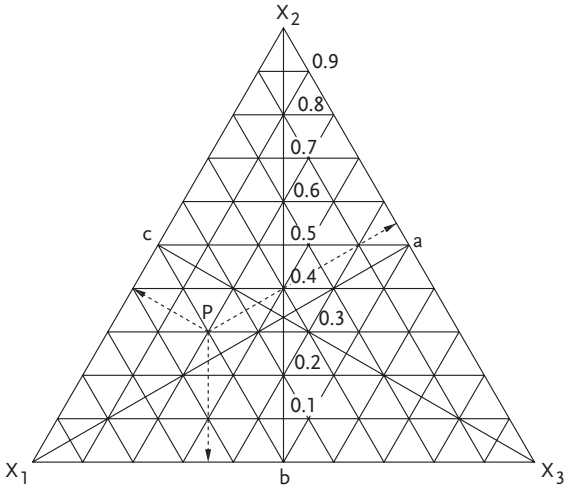

Figure 3.7 Gibbs's concentration triangle

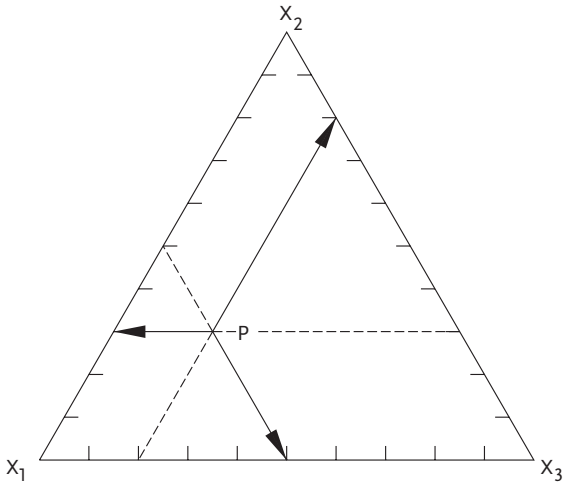

Figure 3.8 Rozebum's concentration triangle

In the concentration triangle, points lying on a straight line originating from a vertex correspond to mixtures with a constant ratio of components represented by the other two vertices. The property (y) is normally thought of as projections of lines of constant value on the plane of the concentration triangle.

At q=4, the regular simplex is a tetrahedron where each vertex represents a straight component, an edge represents a binary system, and a face a ternary one. Points inside the tetrahedron correspond to quaternary systems.

It is known that to construct multicomponent *mixture* diagrams we have, as a rule, to do a large number of experiments. *Scheffe* [5] suggested in 1958 that to solve such problems we should use some properties of geometric figures that are generally used, as demonstrated, to illustrate mixture compositions. *Kurnakov* [11] has also demonstrated that a composition of a q-dimensional mixture may be given by a q-1 dimensional simplex. It is also known [11] that to each phase or a complex of phases, which are in balance in such a system, corresponds a definite geometric interpretation or function (principle of coincidence) whereby the function is continuous (principle of continuity). It is also clear that any continuous function may be approximated or developed into *Taylor's* order. Hence, a change in property of a mixture may be expressed by a polynomial of definite degree, by way of independent variables X_1, X_2,...,X_q where X_i is the ratio of the i-th component in the mixture. A polynomial of n-th degree with q variables has $\binom{n}{q+n}$ coefficients:

$$\widehat{y} = b_0 + \sum_{1\le i\le q} b_i X_i + \sum_{1\le i\le j\le q} b_{ij} X_i X_j + \sum_{1\le i\le j\le k\le q} b_{ijk} X_i X_j X_k + \ldots + \sum b_{i1,i2,\ldots,in} X_{i1} X_{i2} \ldots X_{in} \tag{3.11}$$

Scheffe [5] suggested to describe mixture properties by *reduced polynomials* obtainable from Eq. (3.11), which is subject to the normalization condition of Eq. (3.2) for a sum of independent variables. We shall demonstrate below how, for instance, such a reduced second-degree polynomial is derived for a ternary system. The polynomial has the general form:

$$\widehat{y} = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \tag{3.12}$$

Since:

$$X_1+X_2+X_3=1 \tag{3.13}$$

We have:

$$b_0X_1+b_0X_2+b_0X_3=b_0 \tag{3.14}$$

Multiplying Eq. (3.13) by X_1, X_2 and X_3 in succession gives:

$$\begin{aligned} X_1^2 &= X_1 - X_1X_2 - X_1X_3 \\ X_2^2 &= X_2 - X_1X_2 - X_2X_3 \\ X_3^2 &= X_3 - X_1X_3 - X_2X_3 \end{aligned} \tag{3.15}$$

Substituting Eqs. (3.14) and (3.15) into (3.12), we obtain after necessary transformations:

$$\widehat{y} = (b_0 + b_1 + b_{11})X_1 + (b_0 + b_2 + b_{22})X_2 + (b_0 + b_3 + b_{33})X_3 + (b_{12} - b_{11} - b_{22})X_1X_2 + +(b_{13} - b_{11} - b_{33})X_1X_3 + (b_{23} - b_{22} - b_{33})X_2X_3 \tag{3.16}$$

We denote

$$\beta_i = b_0 + b_i + b_{ii}; \beta_{ij} = b_{ij} - b_{ii} - b_{jj} \tag{3.17}$$

Then we arrive at the reduced second-degree polynomial in three variables:

$$\widehat{y} = \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 \tag{3.18}$$

Thus the number of coefficients has decreased from ten to six. In a general case for a q-component system different degrees of regression models have these forms:

a) Linear regression model:

$$\widehat{y} = \sum_{1\le i\le q} \beta_iX_i \tag{3.19}$$

b) Square regression model:

$$\widehat{y} = \sum_{1\le i\le q} \beta_iX_i + \sum_{1\le i\prec j\le q} \beta_{ij}X_iX_j \tag{3.20}$$

c) Incomplete cube regression model:

$$\widehat{y} = \sum_{1\le i\le q} \beta_iX_i + \sum_{1\le i\prec j\prec\le q} \beta_{ij}X_iX_j + \sum_{1\le i\prec j\prec k\le q} \beta_{ijk}X_iX_jX_k \tag{3.21}$$

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d) Complete cube regression model:

$$\widehat{y} = \sum_{1\le i\le q} \beta_i X_i + \sum_{1\le i\prec j\le q} \beta_{ij} X_i X_j + \sum_{1\le i\prec j\le q} \gamma_{ij} X_i X_j (X_i - X_j) + \sum_{1\le i\prec j\prec k\le q} \beta_{ijk} X_i X_j X_k \tag{3.22}$$

e) Four-degree regression model:

$$\widehat{y} = \sum_{1\le i\le q} \beta_i X_i + \sum_{1\le i\prec j\le q} \beta_{ij} X_i X_j + \sum_{1\le i\prec j\le q} \gamma_{ij} X_i X_j (X_i - X_j) + \sum_{1\le i\prec j\le q} \delta_{ij} X_i X_j (X_i - X_j)^2 + \sum_{1\le i\prec j\prec k\le q} \beta_{iijk} X_i^2 X_j X_k + \sum_{1\le i\prec j\prec k\le q} \beta_{ijjk} X_i X_j^2 X_k + \sum_{\le i\prec j\prec k\le q} \beta_{ijkk} X_i X_j X_k^2 + \sum_{1\le i\prec j\prec k\prec \ell\le q} \beta_{ijk\ell} X_i X_j X_k X_\ell \tag{3.23}$$

3.3 Scheffe Simplex Lattice Design

The most frequently used mixture-"composition-property" designs of experiments belong to *simplex-lattice* designs suggested by *Scheffe* [5]. The basis of this kind of designing experiments is a uniform scatter of experimental points on the so-called simplex lattice. Points, or design points form a [q,n] lattice in a (q-1) simplex, where q is the number of components in a composition and n is the degree of a polynomial. For each component there exist (n+1) similar levels X_i=0,1/n,2/n,...,1 and all possible combinations are derived with such values of component concentrations. So, for instance, for the quadratic lattice [q,2] approximating the response surface with second-degree polynomials (n=2) the following levels of every factor must be used: 0, 1/2 and 1; for the cubic (n=3): 0, 1/3, 2/3 and 1, etc. Number of design points to be performed for obtaining a definite order for a definite number of components is shown in Table 3.13. Some of [3,n] and [4,n] lattices are depicted in Figs. 3.9 and 3.10.

Table 3.13 Number of design points of simplex lattice designs

Number of components	Polynome degree			
	2	Incomplete 3	3	4
3	6	7	10	15
4	10	14	20	35
5	15	25	35	70
6	21	41	56	126
8	36	92	120	330
10	55	175	220	715

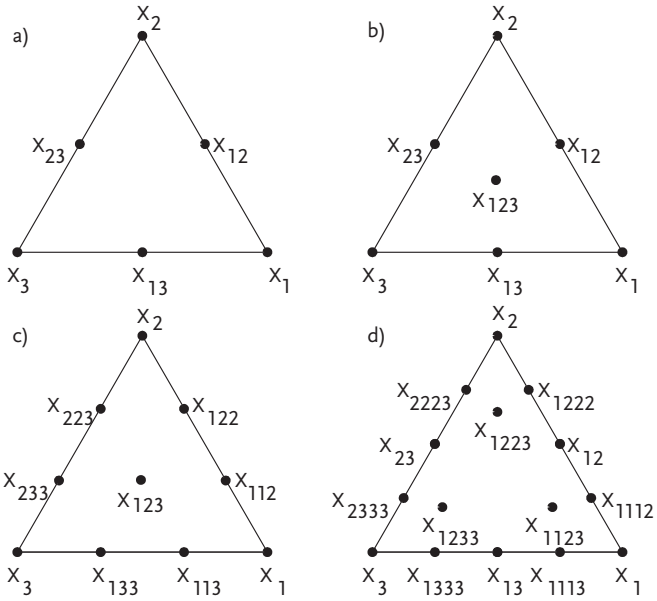

Figure 3.9 {3,n}-lattices: a) for a second-degree polynomial, b) for an incomplete-third-degree polynomial, c) for a third-degree polynomial, d) for a fourth-degree polynomial

When using simplex lattice designs, we apply the principle of upgrading/augmenting, which was analyzed in previous chapters. An increase in polynomial degree is achieved in a standard way after checking lack of fit of the obtained regression model. The rule is known: the higher the polynomial degree, the greater the number of design points in a design of experiments matrix. The required number of design points is calculated from this expression:

$$N = \frac{(N+q-1)!}{n!(q-1)} \tag{3.24}$$

where:

n-is polynomial degree,

q-is number of components.

Results of such calculations are given in Table 3.13. To obtain a linear regression model, one has to do three design points in simplex vertices. To check lack of fit of the model, one has to do an additional design point in the simplex center. The design of the experiment is given in Table 3.14.

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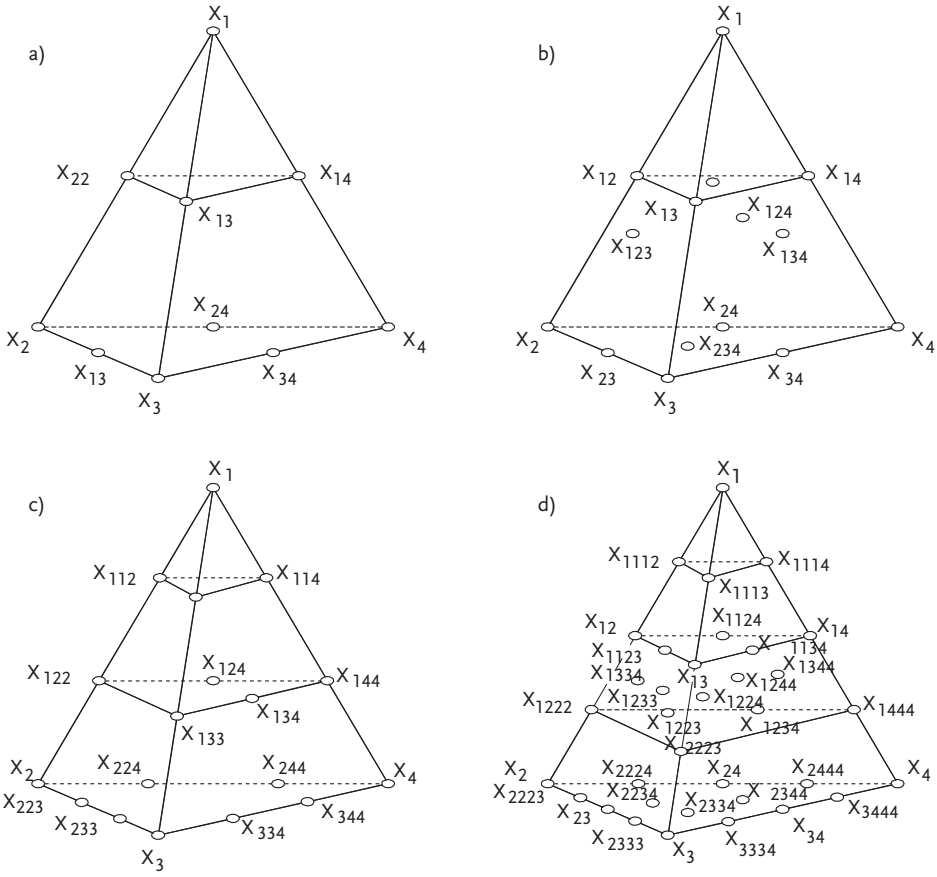

Figure 3.10 {4,n}-lattices: a) for a second-degree polynomial, b) for an incomplete-third-degree polynomial, c) for a third-degree polynomial, d) for a fourth-degree polynomial

Table 3.14 Simplex lattice design {3,1}

N	X_1	X_2	X_3	y
1	1	0	0	y_1
2	0	1	0	y_2
3	0	0	1	y_3
4*	1/3	1/3	1/3	y_{123}

*-Control point

As one may conclude from the above table, subscripts of the mixture property-response symbols indicate the relative proportion of each of the components in the mixture. For example, the mixture 1 (Table 3.14) contains the component X_1 alone,

the property-response of this mixture is denoted by y_1, mixture 4 includes $(1/3)X_1$, $(1/3)X_2$ and $(1/3)X_3$, the property being designated as y_{123}. The design matrix for the simplex lattice {3.3} is shown in the tables below.

Table 3.15 Simplex lattice design {3;2}

No. of trials-N	X_1	X_2	X_3	y
1	1	0	0	y_1
2	0	1	0	y_2
3	0	0	1	y_3
4	0.5	0.5	0	y_{12}
5	0.5	0	0.5	y_{13}
6	0	0.5	0.5	y_{23}
7*	1/3	1/3	1/3	y_{123}

Table 3.16 Simplex lattice design {3;3}

No. of trials-N	X_1	X_2	X_3	y
1	1	0	0	y_1
2	0	1	0	y_2
3	0	0	1	y_3
4	2/3	1/3	0	y_{112}
5	1/3	2/3	0	y_{122}
6	0	2/3	1/3	y_{223}
7	0	1/3	2/3	y_{233}
8	2/3	0	1/3	y_{113}
9	1/3	0	2/3	y_{133}
10	1/3	1/3	1/3	y_{123}

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The number of trials of simplex lattice designs, which depend on the number of components and the degree of regression model, is given Table 3.18.

Table 3.17 Simplex lattice design {3;4}

No. trials	X_1	X_2	X_3	y
1	1	0	0	y_1
2	0	1	0	y_2
3	0	0	1	y_3
4	1/2	1/2	0	y_{12}
5	1/2	0	1/2	y_{13}
6	0	1/2	1/2	y_{23}
7	3/4	1/4	0	y_{1112}
8	1/4	3/4	0	y_{1222}
9	3/4	0	1/4	y_{1113}
10	1/4	0	3/4	y_{1333}
11	0	3/4	1/4	y_{2223}
12	0	1/4	3/4	y_{2333}
13	1/2	1/4	1/4	y_{1123}
14	1/4	1/2	1/4	y_{1223}
15	1/4	1/4	1/2	y_{1233}
16*	1/3	1/3	1/3	y_{123}

Table 3.18 Number of design points of simplex lattice designs

Regression model	Response subscripts	Number of components in mixture				
		3	**4**	**5**	**6**	**7**
Second degree	i	3	4	5	6	7
	ij	3	6	10	15	21
Incomplete third degree	i	3	4	5	6	7
	ij	3	6	10	15	21
	ijk	1	4	10	20	35
Third degree	i	3	4	5	6	7
	iij	6	12	20	30	42
	ijk	1	4	10	20	35
Fourth	i	3	4	5	6	7
	ij	3	6	10	15	21
	iiij	6	12	20	30	42
	ijkl	-	1	5	15	35
	iijk	3	12	30	60	105

Coefficients of regression equations are determined from experimental outcomes in very simple relations. For example, a second degree regression model (3.18) for the case X_1=1; X_2=0 and X_3=0 is brought down to $y_1=\beta_1$. We get $y_2=\beta_2$ and $y_3=\beta_3$ in the same way. For X_1=1/2; X_2=1/2 and X3=0 we get:

$\gamma_{12} = 1/2\beta_1 + 1/2\beta_2 + 1/4\beta_{12} \Rightarrow \beta_{12} = 4\gamma_{12} - 2\gamma_1 - 2\gamma_2;\ \beta_{13} = 4\gamma_{13} - 2\gamma_1 - 2\gamma_3;$
$\beta_{23} = 4\gamma_{23} - 2\gamma_2 - 2\gamma_3$

In a general case for a q-component system, second-order regression coefficients (3.20) are calculated by these relations:

$\beta_i = y_i;\quad \beta_{ij} = 4y_{ij} - 2y_i - 2y_j$

Regression coefficients in other regression models are determined in a similar way:

a) Linear regression model

$$\widehat{y} = \sum_{1 \le i \le q} \beta_i X_i$$
$$\beta_i = \gamma_i \tag{3.25}$$

b) Second-degree regression model

$$\widehat{y} = \sum_{1 \le i \le q} \beta_i X_i + \sum_{1 \le i \prec j \le q} \beta_{ij} X_i X_j$$
$$\beta_i = \gamma_i$$
$$\beta_{ij} = 4\gamma_{ij} - 2\gamma_i - 2\gamma_j \tag{3.26}$$

c) Incomplete third-degree regression model

$$\widehat{y} = \sum_{1 \le i \le q} \beta_i X_i + \sum_{1 \le i \prec j \le q} \beta_{ij} X_i X_j + \sum_{1 \le i \prec j \prec k \le q} \beta_{ijk} X_i X_j X_k$$
$$\beta_i = \gamma_i$$
$$\beta_{ij} = 4\gamma_{ij} - 2\gamma_i - 2\gamma_j \tag{3.27}$$
$$\beta_{ijk} = 27\gamma_{ijk} - 12(\gamma_{ij} + \gamma_{ik} + \gamma_{jk}) + 3(\gamma_i + \gamma_j + \gamma_k)$$

d) Complete third-degree regression model

$$\widehat{y} = \sum_{1 \le i \le q} \beta_i X_i + \sum_{1 \le i \prec j \le q} \beta_{ij} X_i X_j + \sum_{1 \le i \prec j \le q} \gamma_{ij} X_i X_j (X_i - X_j) + \sum_{1 \le i \prec j \le q} \beta_{ijk} X_i X_j X_k$$
$$\beta_i = \gamma_i$$
$$\beta_{ij} = 9/4\left(\gamma_{iij} + \gamma_{ijj} - \gamma_i - \gamma_j\right)$$
$$\gamma_{ij} = 9/4\left(3\gamma_{iij} - 3\gamma_{ijj} - \gamma_i + \gamma_j\right) \tag{3.28}$$
$$\beta_{ijk} = 27\gamma_{ijk} - 27/4\left(\gamma_{iij} + \gamma_{ijj} + \gamma_{iik} + \gamma_{ikk} + \gamma_{jjk} + \gamma_{jkk}\right) + 9/2\left(\gamma_i + \gamma_j + \gamma_k\right)$$

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e) Fourth-degree regression model

$$\widehat{y} = \sum_{1\le i\le q}\beta_i X_i + \sum_{1\le i\prec j\le q}\beta_{ij}X_jX_i + \sum_{1\le i\prec j\le q}\gamma_{ij}X_iX_j\left(X_i - X_j\right) + \sum_{1\le i\prec j\le q}\delta_{ij}X_iX_j\left(X_i - X_j\right)^2 + \sum_{1\le i\prec j\prec k\le q}\beta_{iijk}X_i^2X_jX_k + \sum_{1\le i\prec j\prec k\le q}\beta_{ijjk}X_iX_j^2X_k + \sum_{1\le i\prec j\prec k\le q}\beta_{ijkk}X_iX_jX_k^2 + \sum_{1\le i\prec j\prec k\prec \ell\le q}\beta_{ijk\ell}X_iX_jX_kX_\ell \tag{3.29}$$

$$\beta_i = y_i$$

$$\beta_{ij} = 4y_{ij} - 2y_i - 2y_j$$

$$\gamma_{ij} = (8/3)\left(-y_i + 2y_{iij} - 2y_{ijj} + y_j\right)$$

$$\delta_{ij} = (8/3)\left(-y_i + 4y_{iij} - 6y_{ij} + 4y_{ijj} - y_j\right)$$

$$\beta_{iijk} = 32\left(3y_{iijk} - y_{ijjk} - y_{ijkk}\right) + (8/3)\left(6y_i - y_j - y_k\right) - 16\left(y_{ij} + y_{ik}\right) - (16/3)\left(5y_{iij} + 5y_{iik} - 3y_{ijj} - 3y_{ikk} - y_{jjk} - y_{jkk}\right)$$

$$\beta_{ijjk} = 32\left(3y_{ijjk} - y_{iijk} - y_{ijkk}\right) + (8/3)\left(6y_j - y_i - y_k\right) - 16\left(y_{ij} + y_{jk}\right) - (16/3)\left(5y_{ijj} + 5y_{jjk} - 3y_{iij} - 3y_{jkk} - y_{iik} - y_{ikk}\right)$$

$$\beta_{ijjk} = 32\left(3y_{ijkk} - y_{iijk} - y_{ijjk}\right) + (8/3)\left(6y_k - y_i - y_j\right) - 16\left(y_{ik} + y_{jk}\right) - (16/3)\left(5y_{ikk} + 5y_{jkk} - 3y_{iik} - 3y_{jjk} - y_{iij} - y_{ijj}\right)$$

$$\beta_{ijk\ell} = 256y_{ijk\ell} - 32\left(y_{iijk} + y_{iij\ell} + y_{iik\ell} + y_{ijjk} + y_{ijj\ell} + y_{jjk\ell} + y_{ijkk} + y_{ikk\ell} + y_{jkk\ell} + y_{ij\ell\ell} + y_{jkk\ell} + y_{ik\ell\ell}\right) + (32/3)\left(y_{iij} + y_{iik} + y_{iii\ell} + y_{ijj} + y_{jjk} + y_{jj\ell} + y_{ikk} + y_{jkk} + y_{kkk\ell} + y_{i\ell\ell\ell} + y_{j\ell\ell\ell} + y_{k\ell\ell\ell}\right) \tag{3.30}$$

After obtaining a suitable regression model, a check of lack of fit follows according to the mathematical theory of experiments. Since simplex-lattice designs are saturated, we need degrees of freedom to check lack of fit. To overcome the given problem, we add up additional design points, so-called *control points,* to simplex lattice design, where lack of fit of the model is checked. The number of control points and domain of factor space where those are placed depend on experimental situation, complexity of trials, price, etc. The recommendation is to do control points in:

- the domain of the studied mixture diagram that is interesting for the researcher;
- the existing points;
- the points that are used for obtaining a higher-order model.

The confidence of the response estimate by a regression model differs in different simplex points. Let the variance of response estimate be $S^2_{\hat{y}}$, and let the variance of replicated design points be $S_y{}^2$. An example of determining a variance of response estimate is demonstrated on a second-order regression model for a three-component mixture.

In our reasoning we assume that (1) X_i can be observed without errors, (2) the replication variance is similar at all the design points, and (3) response values are the averages of n_i and n_{ij} replicate observations at appropriate points of the simplex. Then the variances of y_i and y_{ij} will be:

$$S^2_{\hat{y}_i} = S^2_y/n_i$$
$$S^2_{\hat{y}} = S^2_y/n_{ij} \tag{3.31}$$

In the reduced second-order polynomial

$$\hat{y}=\beta_1X_1+\beta_2X_2+\beta_3X_3+\beta_{12}X_1X_2+\beta_{13}X_1X_3+\beta_{23}X_2X_3$$

we replace coefficients by their expressions in terms of responses:

$$\beta_i = \bar{y}_i; \beta_{ij} = 4\bar{y}_{ij} - 2\bar{y}_i - 2\bar{y}_j$$

We then obtain

$$\hat{y} = \bar{y}_1X_1 + \bar{y}_2X_2 + \bar{y}_3X_3 + (4\bar{y}_{12} - 2\bar{y}_1 - 2\bar{y}_2)X_1X_2 + (4\bar{y}_{13} - 2\bar{y}_1 - 2\bar{y}_3)X_1X_3$$
$$+(4\bar{y}_{23} - 2\bar{y}_2 - 2\bar{y}_3)X_2X_3 = \bar{y}_1(X_1 - 2X_1X_2 - 2X_1X_3) + \bar{y}_2(X_2 - 2X_1X_2 - 2X_2X_3)$$
$$+\bar{y}_3(X_3 - 2X_1X_3 - 2X_2X_3) + 4\bar{y}_{12}X_1X_2 + 4\bar{y}_{13}X_1X_3 + 4\bar{y}_{23}X_2X_3 \tag{3.32}$$

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Using the condition: $X_1+X_2+X_3=1$ we transform the coefficients at $\bar{y}_i$:

$$X_1 - 2X_1X_2 - 2X_1X_3 = X_1 - 2X_1(X_2 + X_3) = X_1 - 2X_1(1 - X_1) = X_1(2X_1 - 1),$$
etc. (3.33)

so that:

$$\hat{y} = X_1(2X_1 - 1)\bar{y}_1 + X_2(2X_2 - 1)\bar{y}_2 + X_3(2X_3 - 1)\bar{y}_3 + 4X_1X_2\bar{y}_{12} + 4X_1X_3\bar{y}_{13}$$
$$+4X_2X_3\bar{y}_{23} \tag{3.34}$$

Introducing the designation

$$a_i = X_i(2X_i - 1); a_{ij} = 4X_iX_j \tag{3.35}$$

and using Eqs. (3.30) and (3.31), gives the expression for the variance $S^2_{\hat{y}}$:

$$S^2_{\hat{y}} = S^2_y\left(\sum_{1\le i\le q}\frac{a_i^2}{n_i}\sum_{1\le i\prec j\le q}\frac{a_{ij}^2}{n_{ij}}\right) \tag{3.36}$$

For incomplete-third, third- and fourth-degree polynomials the relationships are derived in much the same way. So, for the incomplete-third-degree polynomial:

$$S^2_{\hat{y}} = S^2_y\left(\sum_{1\le i\le q}\frac{b_i^2}{n_i}\sum_{1\le i\prec j\le q}\frac{b_{ij}^2}{n_{ij}} + \sum_{1\le i\prec j\prec k\le q}\frac{b_{ijk}^2}{n_{ijk}}\right) \tag{3.37}$$

where:

$$b_i = \frac{1}{2}X_i\left(6X_i^2 - 2X_i + 1\right) - 3\sum_{j=1}^{q} X_j^2 \tag{3.38}$$

$$b_{ij} = 4X_iX_j\left(3X_i + 3X_j - 2\right) \tag{3.39}$$

$$b_{ijk} = 27X_iX_jX_k \tag{3.40}$$

For the third-degree polynomial:

$$S^2_{\widehat{y}} = S^2_y\left(\sum_{1\le i\le q}\frac{c_i^2}{n_i} + \sum_{1\le i\prec j\le q}\frac{c_{iij}^2}{n_{iij}} + \sum_{1\le i\prec j\le q}\frac{c_{ijj}^2}{n_{ijj}} + \sum_{1\le i\prec j\prec k\le q}\frac{c_{ijk}^2}{n_{ijk}}\right) \tag{3.41}$$

where:

$$c_i = \frac{1}{2}X_i(3X_i - 1)(3X_i - 2) \tag{3.42}$$

$$c_{iij} = \frac{9}{2}X_iX_j(3X_i - 1) \tag{3.43}$$

$$c_{ijj} = \frac{9}{2}X_iX_j\left(3X_j - 1\right) \tag{3.44}$$

$$c_{ijk} = 27X_iX_jX_k \tag{3.45}$$

For the fourth-degree polynomial:

$$S^2_{\widehat{y}} = S^2_y\left(\sum_{1\le i\le q}\frac{d_i^2}{n_i} + \sum_{1\le i\prec j\le q}\frac{d_{ij}^2}{n_{ij}} + \sum_{1\le i\prec j\le q}\frac{d_{iiij}^2}{n_{iiij}} + \sum_{1\le i\prec j\le q}\frac{d_{ijjj}^2}{n_{ijjj}}\right.$$

$$\left. + \sum_{1\le i\prec j\prec k\le q}\frac{d_{iijk}^2}{n_{iijk}} + \sum_{1\le i\prec j\prec k\le q}\frac{d_{ijjk}^2}{n_{ijjk}} + \sum_{1\le i\prec j\prec k\le q}\frac{d_{ijkk}^2}{n_{ijkk}} + \sum_{1\le i\prec j\prec k\prec \ell\le q}\frac{d_{ijk\ell}^2}{n_{ijk\ell}}\right) \tag{3.46}$$

where:

$$\begin{aligned}
d_i &= 1/6X_i(4X_i - 1)(4X_i - 2)(4X_i - 3)\\
d_{ij} &= 4X_iX_j(4X_i - 1)\left(4X_j - 1\right)\\
d_{ijjj} &= 8/3X_iX_j\left(4X_j - 1\right)\left(4X_j - 2\right)\\
d_{iiij} &= 8/3X_iX_j(4X_i - 1)(4X_i - 2)\\
d_{iijk} &= 32X_iX_jX_k(4X_i - 1)\\
d_{ijjk} &= 32X_iX_jX_k\left(4X_j - 1\right)\\
d_{ijkk} &= 32X_iX_jX_k\left(4X_k - 1\right)
\end{aligned} \tag{3.47)– (3.53}$$

If the number of replications at all the points of the design is equal, i.e. $n_i = n_{ij} = n$, then all the relations for $S^2_{\widehat{y}}$ will take the form:

$$S^2_{\widehat{y}} = S^2_y \times \frac{\xi}{n} \tag{3.54}$$

where for the second-degree polynomial:

$$\xi = \sum_{1\le i\le q} a_i^2 + \sum_{1\le i\prec j\le q} a_{ij}^2 \tag{3.55}$$

for the incomplete-third-degree polynomial:

$$\xi = \sum_{1\le i\le q} b_i^2 + \sum_{1\le i\prec j\le q} b_{ij}^2 + \sum_{1\le i\prec j\prec k\le q} b_{ijk}^2 \tag{3.56}$$

for the third-degree polynomial:

$$\xi = \sum_{1\le i\le q} c_i^2 + \sum_{1\le i\prec j\le q} c_{iij}^2 + \sum_{1\le i\prec j\le q} c_{ijj}^2 + \sum_{1\le i\prec j\prec k\le q} c_{ijk}^2 + \sum_{1\le i\prec j\le q} c_{ij}^2 \tag{3.57}$$

and for the fourth-degree polynomial:

$$\xi = \sum_{1\le i\le q} d_i^2 + \sum_{1\le i\prec j\le q} d_{ij}^2 + \sum_{1\le i\prec j\le q} d_{iiij}^2 + \sum_{1\le i\prec j\le q} d_{ijjj}^2 + \sum_{1\le i\prec j\prec k\le q} d_{iijk}^2 + + \sum_{1\le i\prec j\prec k\le q} d_{ijjk}^2 + \sum_{1\le i\prec j\prec k\le q} d_{ijkk}^2 + \sum_{1\le i\prec j\prec k\prec \ell\le q} d_{ijk\ell}^2 \tag{3.58}$$

As can be seen from Eqs. (3.55) to (3.58), ξ depends only on composition, and geometric interpretation for different order models are to be found in the reference literature [12]. Lack of fit of models is checked in each control point by means of *Students*-test:

$$t_R = \frac{\Delta y}{\sqrt{S_{\bar{y}}^2 + S_{\widehat{y}}^2}} = \frac{\Delta y\sqrt{n}}{S_y\sqrt{1+\xi}} \tag{3.59}$$

where:

$\Delta y = \left| y_{eksp.} - y_{cal.} \right|$;

n-is number of replications;

S_y^2 -is error mean square of trial;

ξ-is the coefficient that depends on mixture composition.

The t_R statistic has the Student distribution and is compared with the tabulated value of $t_{\alpha/\ell;f}$,at a level of significance α, where ℓ- is the number of control points and f-is the number of degrees of freedom for the replication variance. The null hypothesis that the equation is adequate is accepted if $t_R<t_T$ for all the control points. It should also be noted that Eq. (3.59) is valid for the same number of replications in each simplex point. The confidence interval for the response value is:

$$\widehat{y} - \Delta\widehat{y} \ge y \le \widehat{y} + \Delta\widehat{y} \tag{3.60}$$

$$\Delta\widehat{y} = t_{\alpha/k;f} \times S_{\widehat{y}} \tag{3.61}$$

where k-is the number of polynomial coefficients determined.

$$\Delta\widehat{y} = t_{\alpha/k;f} \times \frac{S_y}{\sqrt{n}} \times \xi^{0.5} \tag{3.62}$$

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Example 3.9 [13]

The research objective is to define the composition of a three-component mixture: X_1-ozocerite; X_2-paraffin and X_3-Vaseline, so that the melting point is 66–68 [°C] and penetration 16–18. The mixture is obtained by melting and mixing up.

The experiment has been realized by a simplex lattice design matrix for the fourth-degree model. This model has been chosen, for in case a lower model order is adequate, the excessive points become control points.

Table 3.19 Simplex lattice design {3,4}

No.	Factors			Response Mark	Responses					
	X_1	X_2	X_3		y_1	y_2	$\bar{y}$	y_1'	y_2'	$\bar{y}'$
1	1.0	0.0	0.0	y_1	72.5	73.0	72.75	23	22	22.5
2	0.0	1.0	0.0	y_2	54.0	54.5	54.25	8	8	8
3	0.0	0.0	1.0	y_3	57.5	57.0	57.25	156	156	156
4	0.5	0.5	0.0	y_{12}	65.5	66.0	65.75	12	12	12
5	0.5	0.0	0.5	y_{13}	68.5	68.5	68.5	292	305	298.5
6	0.0	0.5	0.5	y_{23}	52.5	52.5	52.5	67	68	67.5
7	0.75	0.25	0.0	y_{1112}	70.0	70.5	70.25	16	17	16.5
8	0.25	0.75	0.0	y_{1222}	56.0	57.0	56.5	11	10	10.5
9	0.75	0.0	0.25	y_{1113}	72.0	71.5	71.75	74	75	74.5
10	0.25	0.0	0.75	y_{1333}	65.0	65.5	65.25	360	360	360
11	0.0	0.75	0.25	y_{2223}	52.5	53.0	52.75	30	26	28
12	0.0	0.25	0.75	y_{2333}	52.5	54.0	53.25	360	360	360
13	0.5	0.25	0.25	y_{1123}	66.0	66.0	66.0	47	42	44.5
14	0.25	0.5	0.25	y_{1223}	60.0	60.0	60.0	42	40	41.0
15	0.25	0.25	0.5	y_{1233}	59.5	60.0	59.75	144	146	145

Testing the component and mixture melting points was done by the *ASTM-D-127* method. Vaseline penetration testing was done by *ASTM-D-937*, and that of ozocerite, paraffin and the mixture by *ASTM-D-1321* method. Calculations by Eq. (3.29) offered these values of regression coefficients:

$$\beta_1 = y_1 = 72.75; \quad \beta_2 = y_2 = 54.25; \quad \beta_3 = y_3 = 57.25;$$

$$\beta_{12} = 4y_{12} - 2y_1 - 2y_2 = 90.0; \quad \beta_{13} = 4y_{13} - 2y_1 - 2y_3 = 14.0;$$

$$\beta_{23} = 4y_{23} - 2y_2 - 2y_3 = -13.0; \quad \gamma_{12} = \frac{8}{3}(-y_1 + 2y_{1112} - 2y_{1222} + y_2) = 24.03;$$

$$\gamma_{13} = \frac{8}{3}(-y_1 + 2y_{1113} - 2y_{1333} + y_3) = -6.68; \gamma_{23} = \frac{8}{3}(-y_2 + 2y_{2223} - 2y_{2333} + y_3) = 5.54;$$

$$\delta_{12} = \frac{8}{3}(-y_1 + 4y_{1112} - 6y_{12} + 4y_{1222} - y_2) = -38.72;$$

$$\delta_{13} = \frac{8}{3}(-y_1 + 4y_{1113} - 6y_{13} + 4y_{1333} - y_3) = 18.69;$$

$$\delta_{23} = \frac{8}{3}(-\gamma_2 + 4\gamma_{2223} - 6\gamma_{23} + 4\gamma_{2333} - \gamma_3) = -6.68;$$

$$\beta_{1123} = 32(3\gamma_{1123} - \gamma_{1223} - \gamma_{1233}) + \frac{8}{3}(6\gamma_1 - \gamma_2 - \gamma_3) - 16(\gamma_{12} + \gamma_{13})$$
$$-\frac{16}{3}(5\gamma_{1112} + 5\gamma_{1113} - 3\gamma_{1222} - 3\gamma_{1333} - \gamma_{2223} - \gamma_{2333}) = -49.58;$$

$$\beta_{1223} = 32(3\gamma_{1223} - \gamma_{1123} - \gamma_{1233}) + \frac{8}{3}(6\gamma_2 - \gamma_1 - \gamma_3) - 16(\gamma_{12} + \gamma_{23})$$
$$-\frac{16}{3}(5\gamma_{1222} + 5\gamma_{2223} - 3\gamma_{1112} - 3\gamma_{2333} - \gamma_{1113} - \gamma_{1333}) = 159.32;$$

$$\beta_{1233} = 32(3\gamma_{1233} - \gamma_{1123} - \gamma_{1223}) + \frac{8}{3}(6\gamma_3 - \gamma_1 - \gamma_2) - 16(\gamma_{13} + \gamma_{23})$$
$$-\frac{16}{3}(5\gamma_{1333} + 5\gamma_{2333} - 3\gamma_{1113} - 3\gamma_{2223} - \gamma_{1112} - \gamma_{1222}) = -145.94;$$

The fourth-order regression model for the melting point has the form:

$$\begin{aligned}\widehat{\gamma} = {} & 72.75X_1 + 54.25X_2 + 57.25X_3 + 9.0X_1X_2 + 14.0X_1X_3 \\ & -13.0X_2X_3 + 24.03X_1X_2(X_1 - X_2) - 66.8X_1X_3(X_1 - X_3) \\ & +5.34X_2X_3(X_2 - X_3) - 38.72X_1X_2(X_1 - X_2)^2 \\ & +18.69X_1X_3(X_1 - X_3)^2 - 6.68X_2X_3(X_2 - X_3)^2 \\ & -49.58X_1^2X_2X_3 + 159.32X_1X_2^2X_3 - 145.94X_1X_2X_3^2\end{aligned} \tag{3.63}$$

Since the obtained regression model adequately describes experimental outcomes, a check was done by a second-order regression model:

$$\widehat{\gamma} = 72.75X_1 + 54.25X_2 + 57.25X_3 + 9.0X_1X_2 + 14.0X_1X_3 - 13.0X_2X_3 \tag{3.64}$$

Check of lack of fit in control point No. 7:

$$\sigma_{\bar{\gamma}}^2 = \frac{1}{N}\sum_{u=1}^{N}\sigma_u^2 = 0.0893; \sigma_u^2 = \frac{1}{n-1}\sum_{k=1}^{n}\left(\gamma_{uk} - \bar{\gamma}\right)^2;$$

f=N(n-1); α=0.05; ℓ=1; n=2.

where:
N-is total number of points in a simplex lattice design, including the control point;
u-is current number of points in a simplex lattice design;
k≤n-is current number of replicated design points in one point of a simplex lattice design.

For control point 7:

$$\xi = \sum a_i^2 + \sum a_{ij}^2; a_i = X_i(2X_i - 1); a_{ij} = 4X_iX_j.$$
$$a_1 = 0.38; a_2 = 0.125; a_3 = 0.0.$$
$$a_{12} = 0.75; a_{13} = 0.0; a_{23} = 0.0 \Rightarrow \xi = 0.7187.$$

Based on the obtained values it follows:

$$S_{\bar{\gamma}}^2 = \frac{S_{\gamma}^2}{n} \times \xi = \frac{0.0893}{2} \times 0.7187 = 0.0321.$$

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The calculated value of melting point from Eq. (3.64) is:

$\widehat{y}_7 = 69.8125$

The experimental value in point 7 is:

$\bar{y} = 70.25$

It follows that:

$$\Delta y_7 = \left|\widehat{y}_7 - \bar{y}_7\right| = 0.44; t_R = \frac{\Delta y_7 \sqrt{n}}{S_{\bar{y}}\sqrt{1+\xi}} = \frac{0.44\sqrt{2}}{0.2988\sqrt{1+0.7187}} = 1.5885;$$

$$f = N(n-1) = 7(2-1) = 7$$

From the table for Students distribution we get:

$t_{0.05;7} = 2.36 \quad ; \quad t_R = 1.5885 \prec t_T = 2.36$

Hence, the regression model (3.64) is adequate. By analogy, an adequate regression model for penetration has also been obtained:

$$\widehat{y}' = 22.5X_1 + 8.0X_2 + 156.0X_3 - 13.00X_1X_2 + 837.00X_1X_3 - 58.0X_2X_3 \qquad (3.65)$$

The desired composition is obtained by solving this simultaneous system of equations:

$$\begin{cases} 66.0 = 72.75X_1 + 54.25X_2 + 57.25X_3 + 9.0X_1X_2 + 14.0X_1X_3 - 13.0X_2X_3 \\ 18.0 = 22.5X_1 + 8.0X_2 + 156.0X_3 - 13.0X_1X_2 + 837.0X_1X_3 - 58.0X_2X_3 \\ 1.0 = X_1 + X_2 + X_3 \end{cases}$$

$X_1 = 0.6342 \quad ; \quad X_2 = 0.3604 \quad ; \quad X_3 = 0.0054$

The obtained composition, or its properties, completely satisfy the requirements.

Example 3.10 [14]

To obtain maximal bulk density or maximal quality of coke, a study has been done by five coal granulations. Coal granulations were obtained by crushing and classification using sieves. The experiment was performed by a simplex lattice design matrix for a third-order regression model. Design points were done by mixing initial coal granulations in ratios as defined by the design of experiment matrix. Bulk density was measured in an *Agroskina* apparatus, by replicating design points five times. Particle-size properties of the initial coal are given in Table 3.20.

Regression coefficients are calculated by expressions (3.28):

Table 3.20 Coal granulation

Size of particles [mm]	6–3	3–1	1–0.5	0.5–0.25	0.25–0
Ratios of masses	X_1	X_2	X_3	X_4	X_5

$$\begin{aligned}\widehat{y} = {} & 658.3X_1 + 674.4X_2 + 627.8X_3 + 616.1X_4 + 639.6X_5 + 147.7X_1X_2 \\ & +431.5X_1X_3 + 551.2X_1X_4 + 675.9X_1X_5 + 160.4X_2X_3 + 350.2X_2X_4 \\ & +518.3X_2X_5 + 106.0X_3X_4 + 374.0X_3X_5 + 204.7X_4X_5 + 76.3X_1X_2(X_1 - X_2) \\ & +129.3X_1X_3(X_1 - X_3) + 416.5X_1X_4(X_1 - X_4) + 538.8X_1X_5(X_1 - X_5) \\ & +86.2X_2X_3(X_2 - X_3) + 235.8X_2X_4(X_2 - X_4)459.0X_2X_5(X_2 - X_5) \\ & +45.1X_3X_4(X_3 - X_4) + 329.8X_3X_5(X_3 - X_5) + 155.0X_4X_5(X_4 - X_5) \\ & +147.8X_1X_2X_3 + 448.3X_1X_2X_4 + 855.8X_1X_4X_5 - 420.2X_1X_3X_4 \\ & +381.9X_1X_3X_5 - 221.3X_1X_4X_5 - 48.2X_2X_3X_4 + 624.2X_2X_3X_5 \\ & -57.0X_2X_4X_5 + 326.2X_3X_4X_5\end{aligned}$$

Table 3.21 Simplex lattice design {5,3}

Response Mark	Mass ratios of components					$\bar{y}$	S_y^2
	X_1	X_2	X_3	X_4	X_5		
y_1	1	0	0	0	0	658.29	9.25
y_2	0	1	0	0	0	674.36	7.84
y_3	0	0	1	0	0	627.78	11.51
y_4	0	0	0	1	0	616.13	4.88
y_5	0	0	0	0	1	639.58	2.49
y_{123}	0.333	0.333	0.333	0	0	741.13	46.76
y_{124}	0.333	0.333	0	0.333	0	782.77	16.49
y_{125}	0.333	0.333	0	0	0.333	828.21	17.43
y_{134}	0.333	0	0.333	0.333	0	739.48	43.63
y_{135}	0.333	0	0.333	0	0.333	820.64	12.80
y_{145}	0.333	0	0	0.333	0.333	788.90	15.80
y_{234}	0	0.333	0.333	0.333	0	706.15	8.97
y_{235}	0	0.333	0.333	0	0.333	787.33	1.45
y_{245}	0	0.333	0	0.333	0.333	760.49	5.75
y_{345}	0	0	0.333	0.333	0.333	716.00	1.03
y_{112}	0.666	0.333	0	0	0	702.12	3.87
y_{113}	0.666	0	0.333	0	0	765.56	8.95
y_{114}	0.666	0	0	0.333	0	797.59	44.10
y_{115}	0.666	0	0	0	0.333	842.17	16.76
y_{223}	0	0.666	0.333	0	0	700.86	3.54
y_{224}	0	0.666	0	0.333	0	750.24	0.93
y_{225}	0	0.666	0	0	0.333	811.95	3.15
y_{334}	0	0	0.666	0.333	0	650.80	36.92
y_{335}	0	0	0.666	0	0.333	739.27	7.62
y_{445}	0	0	0	0.666	0.333	680.92	25.60
y_{122}	0.333	0.666	0	0	0	696.18	22.69

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Table 3.21 (continued)

Response Mark	Mass ratios of components					$\bar{y}$	S_y^2
	X_1	X_2	X_3	X_4	X_5		
y_{133}	0.333	0	0.666	0	0	712.30	34.87
y_{144}	0.333	0	0	0.666	0	721.83	5.94
y_{155}	0.333	0	0	0	0.666	756.11	1.23
y_{233}	0	0.333	0.666	0	0	672.56	59.38
y_{244}	0	0.333	0	0.666	0	695.89	0.98
y_{255}	0	0.333	0	0	0.666	732.35	11.47
y_{344}	0	0	0.333	0.666	0	640.23	23.33
y_{355}	0	0	0.333	0	0.666	694.34	10.04
y_{455}	0	0	0	0.333	0.666	665.77	3.56
$S_y^2=15.17$; $f=35(5-1)=140$							

Check of lack of fit of the obtained regression model is done in additional control points, Table 3.22, since the simplex lattice design is saturated.

Table 3.22 Control design points

Response Marks	Mass ratios of components					Response		S_y^2
	X_1	X_2	X_3	X_4	X_5	$\bar{y}$	$\hat{y}$	
y_{1234}	0.25	0.25	0.25	0.25	0	748.40	755.30	2.34
y_{1235}	0.25	0.25	0.25	0	0.25	825.78	825.64	2.34
y_{1245}	0.25	0.25	0	0.25	0.25	817.31	816.12	2.34
y_{1345}	0.25	0	0.25	0.25	0.25	782.12	782.95	2.34
y_{2245}	0	0.25	0.25	0.25	0.25	757.09	759.77	2.34
y_{2345}	0.20	0.20	0.20	0.20	0.20	797.65	800.33	1.77
y_{1111}	0.15	0.25	0.15	0.20	0.25	798.80	799.44	1.74
y_{1112}	0.10	0.20	0.20	0.25	0.25	783.33	777.08	1.78

A check of lack of fit by relation (3.59) shows that the regression model is adequate with 95% confidence.

Example 3.11

Nine design points in accord with a simplex lattice design for a second-order model was done in researching for the octane number of a three-component mixture of petrol. The outcomes are given in Table 3.23.

Table 3.23 Simplex lattice design {3,2}

Number of trials	Ratios of components			Response		
	X_1	X_2	X_3	y'	y''	$\bar{y}$
1	1.00	0.00	0.00	100.8	100.9	100.85
2	0.00	1.00	0.00	85.2	85.6	85.40
3	0.00	0.00	1.00	86.0	85.0	85.50
4	0.50	0.50	0.00	88.8	89.3	89.05
5	0.50	0.00	0.50	90.3	90.7	90.50
6	0.00	0.50	0.50	85.5	85.4	85.45
7*	0.333	0.333	0.333	88.3	88.8	88.55
8*	0.150	0.595	0.255	86.6	86.8	86.70
9*	0.300	0.490	0.210	87.6	88.1	87.85

* Control points

By using data from Table 3.23 and formulas (3.26) regression coefficients of a second-order model are calculated.

$$\beta_1 = y_1 = 100.85; \beta_2 = y_2 = 85.40; \beta_3 = y_3 = 85.50;$$

$$\beta_{12} = 2(2y_{12} - y_1 - y_2) = 2(289.05 - 100.85 - 85.40) = -16.30;$$

$$\beta_{13} = 2(2y_{13} - y_1 - y_3) = 2(2 \times 90.50 - 100.85 - 85.50) = -10.70;$$

$$\beta_{23} = 2(2y_{23} - y_2 - y_3) = 2(2 \times 85.45 - 85.40 - 85.50) = 0.00$$

The second-order regression model has this form:

$$\widehat{y} = 100.85X_1 + 85.40X_2 + 85.50X_3 - 16.30X_1X_2 - 10.70X_1X_3$$

Check of lack of fit in control point No. 7:

$$\widehat{y}_7 = 100.85 \times 0.333 + 85.40 \times 0.333 + 85.50 \times 0.333 - 16.30 \times 0.333 \times 0.333 - 10.7 \times 0.333 \times 0.333 = 87.58$$

$$\Delta y_7 = \left|\widehat{y}_7 - \bar{y}_7\right| = |88.55 - 87.58| = 0.97$$

$$t_R = 3.16 \succ t_{(0.025;9)} = 2.66$$

The regression model is inadequate. The second-order model may be built up to an incomplete third-order model by including design point No. 7. By using the expression (3.27) we get:

$$\beta_{123}=27 \times 88.55\text{-}12.0(89.05+90.5+85.45)+3(100.85+85.4+85.5)=26.1$$

The incomplete third-order model has the form:

$$\widehat{y} = 100.85X_1 + 85.40X_2 + 85.50X_3 - 10.30X_1X_2 - 10.70X_1X_3 + 26.10X_1X_2X_3$$

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A check of lack of fit in control points No. 8 and 9 showed that the incomplete cube model is adequate. Graphic interpretation of this model as contour lines is shown Fig. 3.11.

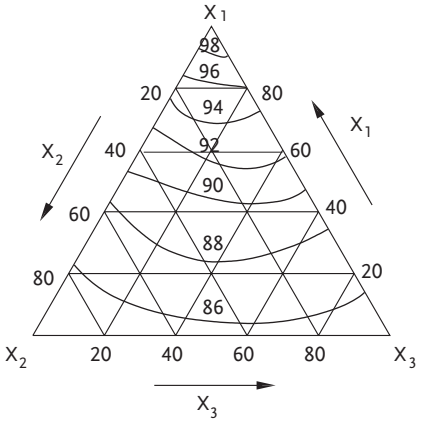

Figure 3.11 Contour plot of a three-component mixture

Example 3.12 [12]

To study the variation of reactivity and porosity of coke with different batch composition, coals of four process groups were analyzed and designated as X_1, X_2, X_3 and X_4. Experiments were conducted on a laboratory pilot plant. The characteristic of coke reactivity was the rate constant of the reaction $C+O_2=2CO$, determined at 1000 °C (y′). The coke porosity was given by the ratio of the true and apparent densities (y″). Assuming that the response surfaces of physical and chemical characteristics of the mixtures at hand can be approximated by polynomials of not very high degree, we shall seek the regression equation in the form of the second-degree polynomial.

In solving the problem, the simplex lattice design {4.2} has been utilized. The second-order design matrix for the quaternary system and experimental results (each trial was repeated twice) are summarized in Table 3.24. By processing these outcomes, the following values of regression coefficients were obtained:

$$\beta_1 = 1.48; \qquad \beta_2 = 0.32; \qquad \beta_3 = 0.50; \qquad \beta_4 = 0.53;$$

$$\beta_{12} = 4y_{12} - 2y_1 - 2y_2 = 4 \times 0.63 - 2 \times 1.48 - 2 \times 0.32 = -1.08;$$

$$\beta_{13} = 4y_{13} - 2y_1 - 2y_3 = 4 \times 0.92 - 2 \times 1.48 - 2 \times 0.50 = -0.22;$$

$$\beta_{14} = 4y_{14} - 2y_1 - 2y_4 = 4 \times 1.08 - 2 \times 1.48 - 2 \times 0.53 = 0.30;$$

$$\beta_{23} = 4y_{23} - 2y_2 - 2y_3 = 4 \times 0.39 - 2 \times 0.32 - 2 \times 0.50 = -0.08;$$

$$\beta_{24} = 4y_{24} - 2y_2 - 2y_4 = 4 \times 0.38 - 2 \times 0.32 - 2 \times 0.53 = -0.18;$$

$$\beta_{34} = 4y_{34} - 2y_3 - 2y_4 = 4 \times 0.54 - 2 \times 0.50 - 2 \times 0.53 = 0.10.$$

Thus the second-order polynomial for the reactivity of the quaternary mixture has the form:

$$\widehat{y}' = 1.48X_1 + 0.32X_2 + 0.50X_3 + 0.53X_4 - 1.08X_1X_2 - 0.22X_1X_3 + 0.3X_1X_4 - 0.08X_2X_3 - 0.18X_2X_4 + 0.1X_3X_4$$

Table 3.24 Simplex lattice design {4,2}

No.	Response	X_1	X_2	X_3	X_4	y′	y″
1	y_1	1	0	0	0	1.48	54.0
2	y_2	0	1	0	0	0.32	55.2
3	y_3	0	0	1	0	0.50	43.3
4	y_4	0	0	0	1	0.53	45.3
5	y_{12}	0.5	0.5	0	0	0.63	53.1
6	y_{13}	0.5	0	0.5	0	0.92	48.0
7	y_{14}	0.5	0	0	0.5	1.08	49.0
8	y_{23}	0	0.5	0.5	0	0.39	46.3
9	y_{24}	0	0.5	0	0.5	0.38	47.1
10	y_{34}	0	0	0.5	0.5	0.54	44.0

For the porosity:

$$\beta_1 = 54.0; \quad \beta_2 = 55.2; \quad \beta_3 = 43.3; \quad \beta_4 = 45.3; \quad \beta_{12} = -6.4;$$
$$\beta_{13} = -2.6; \quad \beta_{14} = -2.6; \quad \beta_{23} = -11.8; \quad \beta_{24} = -12.6; \quad \beta_{34} = -1.2.$$

and the regression equation will be:

$$\widehat{y}'' = 54.0X_1 + 55.2X_2 + 43.3X_3 + 45.3X_4 - 6.4X_1X_2 - 2.6X_1X_3 - 2.6X_1X_4 - 11.8X_2X_3 - 12.6X_2X_4 - 1.2X_3X_4$$

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To test the adequacy of equations derived, 25 test points are used-Table 3.25.

The coordinates of these are selected so that a fourth-degree polynomial can be built, if the regression equations fail to fit adequately. The replication errors are:

$$S_{y''} = 0.075; S_{y''} = 1.5.$$

The number of degrees of freedom was f=35. The number of replicate observations at each point is n=2. At a significant level α=0.05, and f=35, $t_{T(0.05;35)}$=3.6. Thus, both equations are found to adequately fit the experiment.

Table 3.25 Control points

No.	Response	y'	$\widehat{y}$	$\Delta y'$	y''	$\widehat{y}''$	$\Delta y''$	ξ	t_R'	t_R''
1	y_{1112}	0.77	0.99	0.22	53.5	53.1	0.4	0.72	3.16	0.3
2	y_{1113}	1.15	1.19	0.04	51.0	50.8	0.2	0.72	0.575	0.15
3	y_{1114}	1.25	1.20	0.05	50.3	51.3	1.0	0.72	0.72	0.72
4	y_{2223}	0.31	0.34	0.03	49.0	50.1	1.1	0.72	0.43	0.80
5	y_{2224}	0.39	0.34	0.05	52.3	50.4	1.9	0.72	0.72	1.37
6	y_{3334}	0.55	0.52	0.03	45.0	43.6	1.4	0.72	0.43	0.5
7	y_{1222}	0.35	0.41	0.06	57.2	53.8	2.6	0.72	0.86	0.7
8	y_{1333}	0.75	0.7	0.05	44.0	45.5	1.5	0.72	0.72	1.2
9	y_{1444}	0.94	0.82	0.12	48.9	47.0	1.9	0.72	1.72	0.4
10	y_{2333}	0.51	0.44	0.07	43.4	44.0	0.7	0.72	1.0	0.75
11	y_{2444}	0.36	0.45	0.09	46.3	45.4	0.9	0.72	0.74	0.8
12	y_{3444}	0.57	0.50	0.07	44.5	44.6	0.1	0.72	1.34	0.6
13	y_{1123}	0.82	0.77	0.05	52.4	49.8	2.6	0.59	0.74	1.95
14	y_{1124}	0.90	0.81	0.09	51.5	50.1	1.4	0.59	1.3	1.2
15	y_{1134}	1.17	1.00	0.17	47.0	48.4	1.4	0.59	2.54	0.3
16	y_{1223}	0.49	0.51	0.02	50.6	49.5	1.1	0.59	0.3	0.5
17	y_{1224}	0.52	0.53	0.01	48.0	49.8	1.8	0.59	0.15	0.4
18	y_{1334}	0.76	0.75	0.01	46.7	45.8	0.9	0.59	0.15	1.3
19	y_{2234}	0.44	0.40	0.04	48.4	46.5	1.9	0.59	0.6	0.7
20	y_{2334}	0.48	0.46	0.02	46.8	44.4	2.4	0.59	0.3	1.0
21	y_{1233}	0.58	0.63	0.05	45.7	46.7	1.0	0.59	0.74	1.4
22	y_{1244}	0.59	0.68	0.09	47.0	47.6	0.6	0.59	1.34	0.8
23	y_{1344}	0.78	0.80	0.02	49.0	46.4	2.6	0.59	0.3	1.2
24	y_{2344}	0.42	0.44	0.02	46.0	44.8	1.2	0.59	0.3	0.8
25	y_{1234}	0.60	0.65	0.05	48.0	47.0	1.0	0.44	0.78	0.79

3.4 Simplex Centroid Design

Scheffe's simplex centroid designs contain 2^q-1 points, q of which fall on straight components, C_q^2 on binary mixtures, C_q^3 on ternary mixtures, and so forth, and one observation on a q-component mixture. Simplex centroid designs, consist of the points whose coordinates are (1,0,...,0), (1/2, 1/2,0,...,0),...,(1/q,1/q,...,1/q), and of all the points that can be obtained from these by permutations of coordinates. Thus, the design contains a point at the center (centroid) of the simplex and the centroids of all the component simplexes of lesser dimension, its proper faces.

Polynomials obtained from simplex-centroid designs contain as many coefficients as there are points in the design, and for the q-component mixture they have the form:

$$\widehat{y} = \sum_{1\le i\le q} \beta_i X_i + \sum_{1\le i\le j\le q} \beta_{ij} X_i X_j + \sum_{1\le i\prec j\prec k\le q} \beta_{ijk} X_i X_j X_k + \beta_{12\ldots q} X_1 X_2 \ldots X_q \qquad (3.66)$$

For a given number of components q, there exists only one simplex-centroid design. The simplex-lattice design intended to derive the polynomial of incomplete third-degree is the simplex-centroid design for a ternary mixture (Fig. 3.10b). By way of example, we build the simplex-centroid design for a quaternary system (q=4). The number of observations in the design is $N=2^q-1=2^4-1=15$. The arrangement of points over the concentration tetrahedron is shown in Fig. 3.10c, and the respective simplex-centroid design is shown in Table 3.26.

Table 3.26 Matrix of simplex centroid design for a quaternary system q=4

N	X_1	X_2	X_3	X_4	y	N	X_1	X_2	X_3	X_4	y
1	1	0	0	0	y_1	**9**	0	1/2	0	1/2	y_{24}
2	0	1	0	0	y_2	**10**	0	0	1/2	1/2	y_{34}
3	0	0	1	0	y_3	**11**	1/3	1/3	1/3	0	y_{123}
4	0	0	0	1	y_4	**12**	1/3	1/3	0	1/3	y_{124}
5	1/2	1/2	0	0	y_{12}	**13**	1/3	0	1/3	1/3	y_{134}
6	1/2	0	1/2	0	y_{13}	**14**	0	1/3	1/3	1/3	y_{234}
7	1/2	0	0	1/2	y_{14}	**15**	1/4	1/4	1/4	1/4	y_{1234}
8	0	1/2	1/2	0	y_{23}						

The polynomial of Eq. (3.67) for q=4 includes 15 terms and has the form:

$$\widehat{y} = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{14} X_1 X_4 + \beta_{23} X_2 X_3 + \beta_{24} X_2 X_4 + \beta_{34} X_3 X_4 + \beta_{123} X_1 X_2 X_3 + \beta_{124} X_1 X_2 X_4 + \beta_{134} X_1 X_3 X_4 + \beta_{234} X_2 X_3 X_4 + \beta_{1234} X_1 X_2 X_3 X_4 \quad (3.67)$$

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Making recourse to the saturation property of the design and substituting in succession the coordinates of experimental points 1 through 15 into the polynomial of Eq. (3.67), we determine the polynomial coefficients:

$$\beta_1 = y_1 \quad ; \quad \beta_2 = y_2 \quad ; \quad \beta_3 = y_3 \quad ; \quad \beta_4 = y_4; \quad (3.68)$$

$$\begin{aligned} \beta_{12} &= 4y_{12} - 2y_1 - 2y_2; \\ \beta_{13} &= 4y_{13} - 2y_1 - 2y_3; \\ \beta_{14} &= 4y_{14} - 2y_1 - 2y_4; \\ \beta_{23} &= 4y_{23} - 2y_2 - 2y_3; \\ \beta_{24} &= 4y_{24} - 2y_2 - 2y_4; \\ \beta_{34} &= 4y_{34} - 2y_3 - 2y_4; \end{aligned} \quad (3.69)$$

$$\beta_{123} = 27\gamma_{123} - 12(\gamma_{12} + \gamma_{13} + \gamma_{23}) + 3(\gamma_1 + \gamma_2 + \gamma_3)$$
$$\beta_{124} = 27\gamma_{124} - 12(\gamma_{12} + \gamma_{14} + \gamma_{24}) + 3(\gamma_1 + \gamma_2 + \gamma_4)$$
$$\beta_{134} = 27\gamma_{134} - 12(\gamma_{13} + \gamma_{14} + \gamma_{34}) + 3(\gamma_1 + \gamma_3 + \gamma_4) \quad (3.70)$$
$$\beta_{234} = 27\gamma_{234} - 12(\gamma_{23} + \gamma_{24} + \gamma_{34}) + 3(\gamma_2 + \gamma_3 + \gamma_4)$$

$$\beta_{1234} = 256\gamma_{1234} - 108(\gamma_{123} + \gamma_{124} + \gamma_{134} + \gamma_{234}) + 32(\gamma_{12} + \gamma_{13} + \gamma_{14} + \gamma_{23} + \gamma_{24} + \gamma_{34}) - 4(\gamma_1 + \gamma_2 + \gamma_3 + \gamma_4) \quad (3.71)$$

In a similar manner, for the polynomial of Eq. (3.66), for the q-component mixture, regression coefficients are calculated as follows:

$$\beta_i = \gamma_i \quad (3.72)$$

$$\beta_{ij} = 4\gamma_{ij} - 2\gamma_i - 2\gamma_j = 2\left[2\gamma_{ij} - \left(\gamma_i + \gamma_j\right)\right] \quad (3.73)$$

$$\beta_{ijk} = 27\gamma_{ijk} - 12\left(\gamma_{ij} + \gamma_{ik} + \gamma_{jk}\right) + 3\left(\gamma_i + \gamma_j + \gamma_k\right) = 3\left[9\gamma_{ijk} - 4\left(\gamma_{ij} + \gamma_{ik} + \gamma_{jk}\right) + \left(\gamma_i + \gamma_j + \gamma_k\right)\right] \quad (3.74)$$

$$\beta_{ijkm} = 256\gamma_{ijkm} - 108\left(\gamma_{ijk} + \gamma_{ijm} + \gamma_{ikm} + \gamma_{jkm}\right) + 32\left(\gamma_{ij} + \gamma_{ik} + \gamma_{im} + \gamma_{jk} + \gamma_{jm} + \gamma_{km}\right) - 4\left(\gamma_i + \gamma_j + \gamma_k + \gamma_m\right) = 4\left[64\gamma_{ijkm} - 27\left(\gamma_{ijk} + \gamma_{ijm} + \gamma_{ikm}\right) + 8\left(\gamma_{ij} + \gamma_{ik} + \gamma_{im} + \gamma_{jk} + \gamma_{jm} + \gamma_{km}\right) - \left(\gamma_i + \gamma_j + \gamma_k + \gamma_m\right)\right] \quad (3.75)$$

In the general case, the formula for coefficients of regression equation obtained from the simplex-centroid design, takes the form [6]:

$$\beta_{ij\bullet\bullet\bullet} = r\sum_{t}^{r}(-1)^{r-t}t^{r-1}S_t \quad (3.76)$$

where:
r-is the number of indices at coefficients β_{ij};
S_t-is the sum of experimental results for all the t-component mixtures taken in equal proportions (1/t).

For example, for the β_{ijk} coefficients we have r=3(i, j, k) and three sums:

$y_i+y_j+y_k=S_1$ 1/t=1 (3.77)

$y_{ij}+y_{jk}+y_{ik}=S_2$; 1/t=1/2 (3.78)

$y_{ijk}=S_3$; 1/t=1/3 (3.79)

Thus:

$$\beta_{ijk} = 3\left[(-1)^{3-1}1^{3-1}S_1 + (-1)^3 + (-1)^{3-2}\times 2^{3-1}S_2 + (-1)^{3-3}3^{3-1}S_3\right] = 3\left[\left(\gamma_i + \gamma_j + \gamma_k\right) - 4\left(\gamma_{ij} + \gamma_{ik} + \gamma_{jk}\right) + 9\gamma_{ijk}\right] \quad (3.80)$$

Adequacy of a regression equation derived by the simplex-centroid design is tested and the confidence intervals of property values, predicted by the equation, are assigned in much the same way as in the case of the simplex-lattice method.

Example 3.13 [12]

We seek to determine how the activity (y_1) and durability (y_2) of a platinum catalyst supported by a nonporous metal carrier depend on the catalyst composition at 350 °C. The total mass of components was maintained constant from experiment to experiment. Taking it to be unity, we can write:

$$\sum_{i=1}^{3} X_i = 1$$

where:

X_1-the component of Pt/Al_2O_3 was a reforming catalyst;

X_2 and X_3-also components, were inorganic oxides of metals belonging to Groups II and III of the periodic table.

The simplex centroid design for q=3 is applied. The design matrix and experimental results are arrayed in Table 3.27.

Table 3.27 Simplex centroid design for q=3

N	X_1	X_2	X_3	y_1	y_2
1	1	0	0	97.4	62
2	0	1	0	3.0	73
3	0	0	1	4.7	47
4	0.5	0.5	0	70.0	64
5	0.5	0	0.5	66.0	55
6	0	0.5	0.5	6.8	72
7	0.333	0.333	0.333	95.4	67

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Using Eq. (3.27) and Table 3.27, the coefficients of regression equations are derived for both the catalyst activity and durability:

$$\widehat{y}_1 = 97.4X_1 + 3.0X_2 + 4.7X_3 + 79.3X_1X_2 + 59.9X_1X_3 + 11.8X_2X_3 + 175.35X_1X_2X_3 \quad (3.81)$$

$$\widehat{y}_2 = 62X_1 + 73X_2 + 47X_3 - 14X_1X_2 + 2X_1X_3 + 48X_2X_3 + 63X_1X_2X_3 \quad (3.82)$$

The replication error in measuring the catalyst activity is S_{y_1}=3.24, and durability S_{y_2}=2.37. The adequacy of the regression equations (3.81) and (3.82) is tested using the Student t-test at the control-test points 8, 9 and 10, Table 3.28.

Table 3.28 Check of lack of fit of regressions - Eqs. (3.81); (3.82)

N	X_1	X_2	X_3	y_1	$\widehat{y}_1$	y_2	$\widehat{y}_2$
8	0.333	0.667	0	46	52	72	66
9	0.667	0.333	0	96	84	63	70
10	0.580	0.320	0.097	91	98	62	65

At all the test-control points, the values of the t-test are less than the tabulated value at the significance level α=0.05. Fig. 3.12 presents the lines of constant catalyst activity and durability plotted from Eqs. (3.81) and (3.82). The greatest activity of the catalyst corresponds to the area where the values of the component X_1>0.4. The durability of 65 per cent appears quite satisfactory. Of greatest interest are the points lying where the equal yield curves for y_2=65[%] and y_1=100[%] intersect. The trial 10 executed within the specified area gave a good agreement (within experimental error) of experimental results with theory.

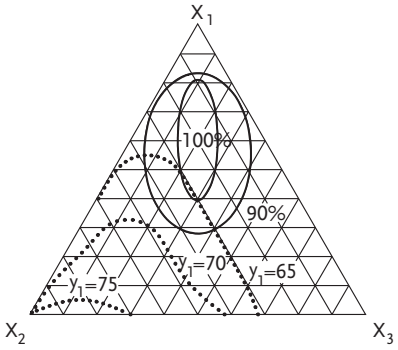

Figure 3.12 Isolinies for y_1 (.....) and y_2 (——)

3.5 Extreme Vertices Designs

It has been explained that when testing mixture diagrams, factor space is usually a regular simplex with q-vertices in a q-1 dimension space. In such a case, the task of mathematical theory of experiments consists of determining in the given simplex the minimum possible number of points where the design points will be done and based on which coefficients of the polynomial that adequately describes system behavior will be determined. This problem, for the case when there are no limitations on ratios of individual components, as presented in the previous chapter, was solved by Scheffe in 1958 [5]. However, a researcher may in practice often be faced with multicomponent mixtures where definite limitations are imposed on ratios of individual components:

$$0 \le a_i \le X_i \le b_i \le 1; \qquad \sum X_i = 1; \qquad i = 1.2,\ldots,q$$

where a_i and b_i correspond to upper and lower limit ratios of the i-th component. This situation occurs when doing experiments in simplex vertices has no physical sense, or when the researcher is only interested in a *local region* of a simplex space. In this, the researched local region is usually a multiangle whose vertices and the center represent the gathering of points of an experimental design. Depending on the number of components, the local region may be:

1. The region studied is a simplex

The local area of interest in the diagram may be an *irregular simplex* with unknown vertex coordinates:

$$A_1\left(X_1^{(1)}, X_2^{(1)}, \ldots, X_q^{(1)}\right), A_2\left(X_1^{(2)}, X_2^{(2)}, \ldots, X_q^{(2)}\right), \ldots, A_q\left(X_1^{(q)}, X_2^{(q)}, \ldots, X_q^{(q)}\right).$$

In order that Scheffes simplex lattice designs may be applied to this case, a renormalization is performed and compositions at vertices A_j(j=1, 2,..., q) are taken to be independent *pseudocomponents* so that for all the range of the local simplex the condition be met:

$$\sum_i^q Z_i = 1 \tag{3.83}$$

The experimental design is in the pseudocomponent coordinates. All the designs discussed earlier can be built in the new variables Z_1, Z_2,..., Z_q that satisfy the condition of Eq. (3.83). To conduct the experiments it is required to convert the pseudocomponents Z_i into the initial components with real ratios X_i. For the u-th design point this conversion is defined by the formula:

$$\begin{aligned} X_1^{(u)} &= X_i^{(1)} + Z_2^{(u)}\left(X_i^{(2)} - X_i^{(1)}\right) + Z_3^{(u)}\left(X_i^{(3)} - X_i^{(1)}\right) \\ &+ \ldots + Z_q^{(u)}\left(X_i^{(q)} - X_i^{(1)}\right) \end{aligned} \tag{3.84}$$

where X_i is the i-th component content of the vertex $Z_j(A_j)$.

After the design has been realized the coefficients of the regression equation are calculated in pseudocomponent coordinates:

$$\widehat{y} = f\left(Z_1, Z_2, \ldots, Z_q\right) \tag{3.85}$$

using earlier relationships for pertinent designs, and then adequacy of fit is tested. For Eq. (3.85) to be applied in practice, it is written in the initial coordinate system using an affine transformation of the form:

$$\left.\begin{aligned} Z_1 &= Z_1^{(1)} + X_2\left(Z_1^{(2)} - Z_1^{(1)}\right) + X_3\left(Z_1^{(3)} - Z_1^{(1)}\right) + \ldots + X_q\left(Z_1^{(q)} - Z_1^{(1)}\right) \\ Z_2 &= Z_2^{(1)} + X_2\left(Z_2^{(2)} - Z_2^{(1)}\right) + X_3\left(Z_2^{(3)} - Z_2^{(1)}\right) + \ldots + X_q\left(Z_2^{(q)} - Z_2^{(1)}\right) \\ \cdot\quad &= \cdot\quad + \cdot\quad\left(\cdot\quad - \cdot\quad\right) + \cdot\quad\left(\cdot\quad - \cdot\quad\right) + \ldots + \cdot\quad\left(\cdot\quad - \cdot\quad\right) \\ Z_{q-1} &= Z_{q-1}^{(1)} + X_2\left(Z_{q-1}^{(2)} - Z_{q-1}^{(1)}\right) + X_3\left(Z_{q-1}^{(3)} - Z_{q-1}^{(1)}\right) + \ldots + X_q\left(Z_{q-1}^{(q)} - Z_{q-1}^{(1)}\right) \end{aligned}\right\} \tag{3.86}$$

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Values of $Z_i^{(j)}$ are calculated solving (q-1) sets of equations below

$$\left.\begin{array}{l} X_1^{(1)} Z_1^{(1)} + X_2^{(1)} Z_1^{(2)} + X_3^{(1)} Z_1^{(3)} + \ldots + X_q^{(1)} Z_1^{(q)} = 1 \\ X_1^{(2)} Z_1^{(1)} + X_2^{(2)} Z_1^{(2)} + X_3^{(2)} Z_1^{(3)} + \ldots + X_q^{(2)} Z_1^{(q)} = 0 \\ \cdots \\ X_1^{(q)} Z_1^{(1)} + X_2^{(q)} Z_1^{(2)} + X_3^{(q)} Z_1^{(3)} + \ldots + X_q^{(q)} Z_1^{(q)} = 0 \end{array}\right\}$$

$$\left.\begin{array}{l} X_1^{(1)} Z_2^{(1)} + X_2^{(1)} Z_2^{(2)} + X_3^{(1)} Z_2^{(3)} + \ldots + X_q^{(1)} Z_2^{(q)} = 0 \\ X_1^{(2)} Z_2^{(1)} + X_2^{(2)} Z_2^{(2)} + X_3^{(2)} Z_2^{(3)} + \ldots + X_q^{(2)} Z_2^{(q)} = 1 \\ \cdots \\ X_1^{(q)} Z_2^{(1)} + X_2^{(q)} Z_2^{(2)} + X_3^{(q)} Z_2^{(3)} + \ldots + X_q^{(q)} Z_2^{(q)} = 0 \end{array}\right\} \qquad (3.87)$$

$$\left.\begin{array}{l} X_1^{(1)} Z_{q-1}^{(1)} + X_2^{(1)} Z_{q-1}^{(2)} + X_3^{(1)} Z_{q-1}^{(3)} + \ldots + X_q^{(1)} Z_{q-1}^{(q)} = 0 \\ X_1^{(2)} Z_{q-1}^{(1)} + X_2^{(2)} Z_{q-1}^{(2)} + X_3^{(2)} Z_{q-1}^{(3)} + \ldots + X_q^{(2)} Z_{q-1}^{(q)} = 0 \\ \cdots \\ X_1^{(q)} Z_{q-1}^{(1)} + X_2^{(q)} Z_{q-1}^{(2)} + X_3^{(q)} Z_{q-1}^{(3)} + \ldots + X_q^{(q)} Z_{q-1}^{(q)} = 1 \end{array}\right\}$$

where:
$Z_i^{(j)}$-is the pseudocomponent;
Z_i-content of vertices of the initial simplex;
$X_i^{(j)}$-is the i-th component content of vertices Z_j (A_j)(j=1, 2,..., q).

As such a coordinate conversion is only possible for equations in independent variables, the initial regression equation shall be transformed eliminating one variable , e.g. the last one, the q-th as follows:

$$Z_q = 1 - \sum_{i=1}^{q-1} Z_i \qquad (3.88)$$

Example 3.14 [13]

The boiling of the ternary mixture H_2O-K_2HPO_4-K_2CO_3 is studied. It is required to define the regression equation for the boiling point y °C on the mixture composition (in per cent). Not all the concentration triangle is covered, but only a subarea of unsaturated solutions at 20 °C, i.e. a local section of the diagram in the form of a triangle with the vertices Z_1(100; 0.0), Z_2(40; 60.0) and Z_3(50; 0.50) Fig. 3.13.

To deduce the regression equation, an extreme-vertices design is performed with pseudocomponents Z_1, Z_2 and Z_3; and the content of initial components is then determined from Eq. (3.84). The regression equations of the second and incomplete third order are found to be inadequate. Using the property of composition of simplex lattice designs, the design matrix is augmented further to yield fourth-order regression equations-Table 3.29. The experimental conditions are expressed in terms of pseudocomponents Z_i and in the natural scale X (per cent). The mean values of temperature measurements are determined from two replicate observations. The replication error is S_y=0.86. The number of degrees of freedom for the error is f=20.

The coefficients of the fourth-order regression equation are calculated by Eq. (3.29) using the property of saturated design matrix. The regression equation in pseudocomponent variables has the form:

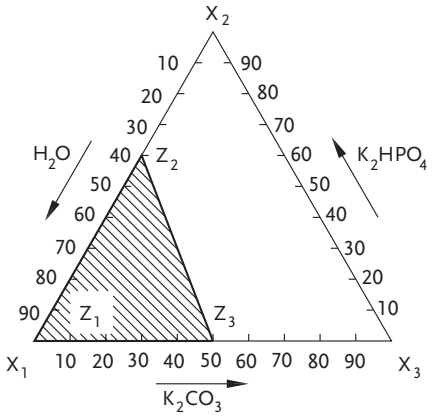

Figure 3.13 Extreme vertices simplex of a three-component mixture: K_2HPO_4-K_2CO_3-H_20

$$\begin{aligned}\widehat{y} = {} & 99.81Z_1 + 113.51Z_2 + 115.69Z_3 - 14.22Z_1Z_2 - 12.13Z_1Z_3 + 0.91Z_2Z_3 \\ & + 6.18Z_1Z_2(Z_1 - Z_2) + 10.12Z_1Z_3(Z_1 - Z_3) - 15.34Z_2Z_3(Z_2 - Z_3) \\ & + 6.90Z_1Z_2(Z_1 - Z_2)^2 - 17.61Z_1Z_3(Z_1 - Z_3)^2 + 6.32Z_2Z_3(Z_2 - Z_3)^2 \\ & + 1.07Z_1^2Z_2Z_3 - 274.61Z_1Z_2^2Z_3 + 142.21Z_1Z_2Z_3^2 \end{aligned} \quad (3.89)$$

Table 3.29 Extreme vertices design

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No. trials	Z_1	Z_2	Z_3	X_1	X_2	X_3	$\bar{y}$
1	1	0	0	100	0	0	99.9
2	0	1	0	40	60	0	113.5
3	0	0	1	50	0	50	115.7
4	0.5	0.5	0	70	30	0	103.1
5	0.5	0	0.5	75	0	25	104.8
6	0	0.5	0.5	45	30	25	114.8
7	0.333	0.333	0.333	63.33	20	16.67	105.6
8	0.75	0.25	0	85	15	0	101.5
9	0.25	0.75	0	55	45	0	107.2
10	0.75	0	0.25	87.5	0	12.5	101.6
11	0.25	0	0.75	62.5	0	37.5	107.7
12	0	0.75	0.25	42.5	45	12.5	112.5
13	0	0.25	0.75	47.5	15	37.5	116.4
14	0.5	0.25	0.25	72.5	15	12.5	103.4
15	0.25	0.5	0.25	57.5	30	12.5	101.4
16	0.25	0.25	0.5	60	15	25	109.0
17*	0.2	0.2	0.6	58	12	30	108.3

Table 3.29 (continued)

No. trials	Z_1	Z_2	Z_3	X_1	X_2	X_3	$\bar{y}$
18*	0.5	0.125	0.375	73.75	7.5	18.75	103.3
19*	0.4	0.15	0.45	68.5	9	22.5	104.2
20*	0.3	0.175	0.525	63.25	10.5	26.25	106.2

Table 3.30 summarizes the results of the regression equation testing for adequacy of fit:

Table 3.30 Control points

No. of trials	$\bar{y}$	$\widehat{y}$	Δy	ξ	t_R
17	108.3	110.7	2.4	1.3	2.16
18	103.3	100.9	2.4	1.0	2.27
19	104.2	107.0	2.8	1.0	2.66
20	106.3	108.7	2.4	1.1	2.16

The tabulated value of Student's t is $t_{T(0.05/4,20)}$=2.8. Equation (3.89) is an adequate fit to the experiment. In Eq. (3.89), we convert from pseudocomponents Z_i to initial variables X_i. For the problem in hand, the sets of simultaneous equations (3.87) take the form:

$$\left.\begin{array}{l} 1Z_1^{(1)} + 0Z_1^{(2)} + 0Z_1^{(3)} = 1 \\ 0.4Z_1^{(1)} + 0.6Z_1^{(2)} + 0Z_1^{(3)} = 0 \\ 0.5Z_1^{(1)} + 0Z_1^{(2)} + 0.5Z_1^{(3)} = 0 \\ 1Z_2^{(1)} + 0Z_2^{(2)} + 0Z_2^{(3)} = 0 \\ 0.4Z_2^{(1)} + 0.6Z_2^{(2)} + 0Z_2^{(3)} = 1 \\ 0.5Z_2^{(1)} + 0Z_2^{(2)} + 0.5Z_2^{(3)} = 0 \end{array}\right\} \tag{3.90}$$

Solving these we obtain:

$$Z_1^{(1)} = 1; \quad Z_1^{(2)} = 0.7; \quad Z_1^{(3)} = -1\ ; \quad Z_2^{(1)} = 0; \quad Z_2^{(2)} = 1.7; \quad Z_2^{(3)} = 0;$$

Substituting the above solutions into Eq. (3.86) we arrive at formulas relating natural coordinates X_i to coordinates Z_i:

$$\left\{\begin{array}{l} Z_1 = 1 - 1.7X_2 - 2X_3 \\ Z_2 = 1.7X_2 \\ Z_3 = 1 - Z_1 - Z_2 = 2X_3 \end{array}\right. \tag{3.91}$$

And, substituting Eq. (3.91) into Eq. (3.89), we get the regression equation in initial coordinates:

$$\begin{aligned} \widehat{y} &= 99.88X_1 + 20.82X_2 - 7.63X_3 + 92.88X_2X_3 - 107.83X_2^2 + 279.28X_3^2 \\ &-1373.69X_2^2X_3 - 243.59X_2X_3^2 + 2230.35X_2^2X_3^2 + 312.78X_2^3 - 965.12X_3^3 \\ &+2146.05X_2^3X_3 - 179.60X_2X_3^3 - 212.96X_2^4 + 1127.1X_3^4 \end{aligned} \tag{3.92}$$

To use the regression equation more conveniently in Fig. 3.14, the isotherms are plotted.

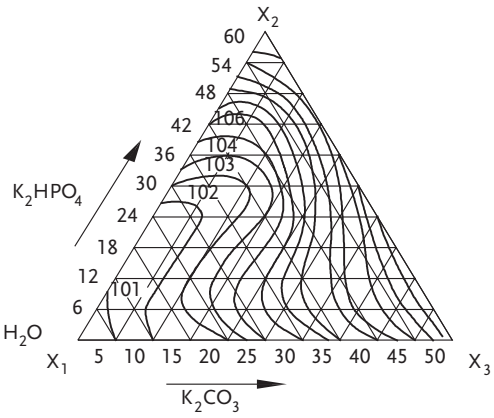

Figure 3.14 Isotherms

2. The space studied is a polyhedron

With constraints on the component concentration variation, in the general case, the space studied forms a polyhedron. In the experimental design we should somehow distribute the points over the polyhedron subject to the condition:

$$0 \le a_i \le X_i \le b_i \le 1 \tag{3.93}$$

This excludes degenerate cases:

$$\sum_{i=1}^{q} a_i \ge 1; \sum_{i=1}^{q} b_i \le 1 \tag{3.94}$$

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As with all previous designs of experiments, the number of design points or trials grows very rapidly (power function) along with the number of factors-components. A need to reduce the number of design points sets up a demand for the remaining points of a design of experiment to cover evenly the local factor space. When the number of components is above five ($q \ge 5$), the calculation of the combinations of experimental conditions of possible design points would include several billion arithmetic operations. Even the fastest IBM computers are, in such a situation unable to do calculations before the time necessary for physical performance of the experiment.

To reduce the scope of calculations and to formalize the approach to the choice of design points of a design of experiment, *McLean* and *Anderson* [16] suggested this procedure:

1. All the possible combinations of the two levels a_i and b_i, are put down for each and every component, but in each combination the content of one component is omitted. The number of these combinations for a q-component mixture is $q \times 2^{q-1}$;

2. Among all the combinations those are selected whose sum of components is less than one and that meet the limitations of Eq. (3.93). Into the combinations selected the omitted components are added in amounts defined by the relationship $\Sigma X_i=1$. The design points thus obtained and satisfying Eq. (3.93) lie at vertices of the bounding polyhedron;
3. To the design points obtained are added center points (centroids) of two-, three-, ..., and (q-1)-dimensional faces of the polyhedron and its center point. Coordinates of a central point are determined by taking average coordinates of previously chosen vertices;
4. Distances between vertices and center of polyhedron are calculated by:

$$d_{ij} = \left[\sum_{r=1}^{q}\left(\frac{X_{ir}-X_{jr}}{b_r-a_r}\right)^2\right]^{0.5} \tag{3.95}$$

5. The point with maximal distance from polyhedron center-point 1, is inserted into the design;
6. The distance between the chosen point 1 and other polyhedron vertices is determined, and the farthest-away vertex becomes part of design of the experiment as point 2;
7. The normed distance $d_{ij}^{'}$ is accepted. The size of the normed distance is smaller if the number of points, necessary to include into the design of experiment, increases. The distance recommended is:

$$d_C^{SR} \le d_{ij}' \le \left(2d_C^{SR}\right)^{0.5} \tag{3.96}$$

where:
d_C^{SR} is the average distance of vertex from center;

8. All previously included points are omitted from the design if their distances from points 1 and 2 is smaller than the accepted norm;
9. From the remaining points, the remotest point from the center (3.96) is included into the design.

This was the way to select vertices, or their coordinates of the local factor space. It should be noted that those are pseudocomponent coordinates.

Example 3.15 [16]
Consider the building of *McLean* and *Anderson's* design for the investigation and optimization of luminance of luminous mixtures, whose components are: X_1-magnesium; X_2-soda; X_3-strontium nitrate and X_4-binder. The mixture composition is subjected to the following constraints:

$0.40 \le X_1 \le 0.60$; $0.10 \le X_2 \le 0.50$; $0.10 \le X_3 \le 0.50$; $0.03 \le X_4 \le 0.08$

Table 3.31 summarizes all the possible combinations of mixture composition with one of the components missing. Accordingly, eight design points, i.e. polyhedron vertices Fig. 3.15 are obtained.

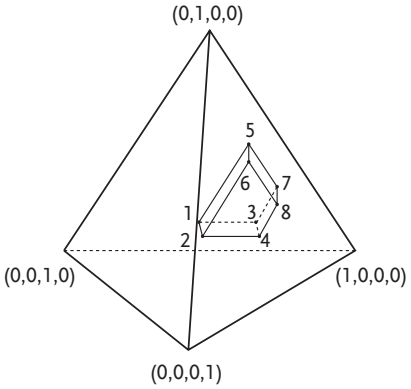

Figure 3.15 Local factor space

Table 3.31 McLean-Anderson design

N	Component content				N	Component content			
	X_1	X_2	X_3	X_4		X_1	X_2	X_3	X_4
1	0.40	0.10	0.10	–	17 (1)	0.40	0.10	0.47*	0.03
2	0.40	0.10	0.50		18 (2)	0.40	0.10	0.42*	0.08
3	0.40	0.50	0.10	–	19	0.40	0.50	–	0.03
4	0.40	0.50	0.50	–	20	0.40	0.50	–	0.08
5	0.60	0.10	0.10	–	21 (3)	0.60	0.10	0.27*	0.03
6	0.60	0.10	0.50	–	22 (4)	0.60	0.10	0.22*	0.08
7	0.60	0.50	0.10	–	23	0.60	0.50	–	0.03
8	0.60	0.50	0.50	–	24	0.60	0.50	–	0.08
9 (5)	0.40	0.47*	0.10	0.03	25	-	0.10	0.10	0.03
10 (6)	0.40	0.42*	0.10	0.08	26	-	0.10	0.10	0.08
11	0.40	–	0.50	0.03	27	-	0.10	0.50	0.03
12	0.40	–	0.50	0.08	28	-	0.10	0.50	0.08
13 (7)	0.60	0.27*	0.10	0.03	29	-	0.50	0.10	0.03
14 (8)	0.60	0.22*	0.10	0.08	30	-	0.50	0.10	0.08
15	0.60	–	0.50	0.03	31	-	0.50	0.50	0.03
16	0.60	–	0.50	0.08	32	-	0.50	0.50	0.08

* Amount of component added

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These points are to be supplemented by the coordinates of center points of all the polyhedron faces, Table 3.32. The coordinates of the polyhedron center point are found by averaging appropriate coordinates of all the eight design vertices; and the centroid coordinates of faces by averaging the coordinates of the points belonging to the face, Table 3.32.

Table 3.32 Selection of face center points in McLean and Anderson's design

N	Component content				Points of face			
	X_1	X_2	X_3	X_4				
(9)	0.50	0.10	0.345	0.055	(1)	(2)	(3)	(4)
(10)	0.50	0.345	0.10	0.055	(5)	(6)	(7)	(8)
(11)	0.40	0.2725	0.2725	0.055	(1)	(2)	(5)	(6)
(12)	0.60	0.1725	0.1725	0.055	(3)	(4)	(7)	(8)
(13)	0.50	0.2350	0.2350	0.030	(1)	(3)	(5)	(7)
(14)	0.50	0.2100	0.2100	0.080	(2)	(4)	(6)	(8)
(15)	0.50	0.2225	0.2225	0.055	Polyhedron center point			

For the quaternary mixture, the design of *McLean* and *Anderson*, together with the experimental results, is provided in Table 3.33.

Table 3.33 McLean-Anderson design for quaternary mixture

N	X_1	X_2	X_3	X_4	y	N	X_1	X_2	X_3	X_4	y
1	0.40	0.10	0.47	0.03	75	**9**	0.50	0.10	0.345	0.055	220
2	0.40	0.10	0.42	0.08	180	**10**	0.50	0.345	0.10	0.055	200
3	0.60	0.10	0.27	0.03	195	**11**	0.40	0.2725	0.2725	0.055	190
4	0.60	0.10	0.22	0.08	300	**12**	0.60	0.1725	0.1725	0.055	310
5	0.40	0.47	0.10	0.03	145	**13**	0.50	0.235	0.235	0.030	200
6	0.40	0.42	0.10	0.08	230	**14**	0.50	0.210	0.210	0.080	410
7	0.60	0.27	0.10	0.03	220	**15**	0.50	0.2225	0.2225	0.055	425
8	0.60	0.22	0.10	0.08	350						

The coefficients of the reduced second-degree polynomial are found by the method of least squares. Here the regression equation will be:

$$\widehat{y} = -1.558X_1 - 2.851X_2 - 2.426X_3 + 14.372X_4 + 8.300X_1X_2 + 8.076X_1X_3 - 6.625X_1X_4 + 3.213X_2X_3 - 16.998X_2X_4 - 17.127X_3X_4 \quad (3.97)$$

As the dependence of the property on components is described adequately by the second-order regression equation, the possibility presented itself to find optimal conditions through the use of nonlinear programming. Subject to the restrictions of Eq. (3.94), the conditions providing the maximum luminance are found to be:

$\widehat{y}_{\max} = 397.48$ for X_1=0.5233; X_2=0.2299; X_3=0.1608; X_4=0.080.

As the number of mixture components increases, the number of points in the design of *McLean-Anderson* grows rapidly. A reduction in the number of observations may be achieved by eliminating some of the face-center points, or by the elimination of points that do not jeopardize the rest being distributed over the space under study more or less uniformly.

Example 3.16 [17]

In a study of ballistic properties of a three-modal composite rocket propellant, the effect of coarse, medium and fine fractions of ammonium perchlorate on burning rate at 70 bar and 25 °C has been mathematically modeled. Limitations were imposed on the ratios of all three granulations of ammonium perchlorate:

x_1-fine fraction AP-7 μm=0.3–0.7;

x_2-coarse fraction AP-400 μm=0.0–0.40;

x_3-medium fraction AP-200 μm=0.30–0.70.

Geometric interpretation of the local factor space is given in Fig. 3.16.

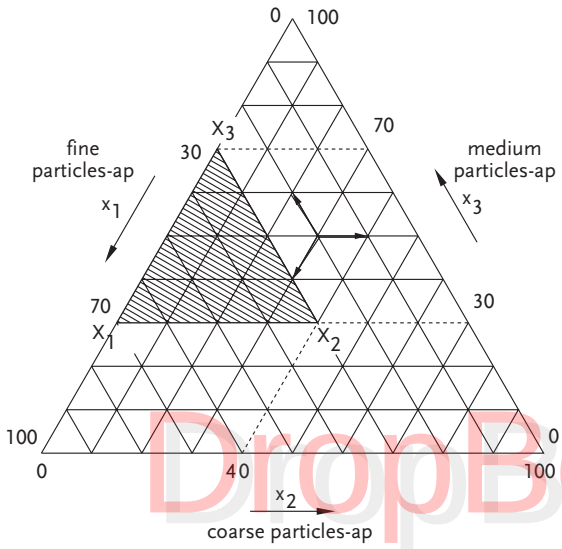

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Figure 3.16 Local factor space

Relations between real and coded ratios of ammonium perchlorate fractions are given by the relations:

$$\begin{cases} x_1 = 0.70X_1 + 0.30X_2 + 0.30X_3 \\ x_2 = 0.40X_2 \\ x_3 = 0.30X_1 + 0.30X_2 + 0.70X_3 \end{cases} \quad (3.98)$$

where:

x_i (i=1, 2, 3)-are real ratios of i-th fraction;

X_i-are coded ratios of i-th fraction.

The design matrix has been defined in accord with the theory of extreme vertices designing of experiments Table 3.34.

Table 3.34 Extreme vertices design

No.	Design matrix			Response marks	Operational matrix			Response y
	X_1	X_2	X_3		x_1	x_2	x_3	
1	1	0	0	y_1	70.00	0.00	30.00	3.92
2	0	1	0	y_2	30.00	40.00	30.00	4.75
3	0	0	1	y_3	30.00	0.00	70.00	4.95
4	1/3	2/3	0	y_{122}	43.33	26.66	30.00	4.25
5	1/3	0	2/3	y_{133}	43.33	0.00	56.66	4.44
6	0	1/3	2/3	y_{233}	30.00	13.13	56.66	4.82
7	2/3	1/3	0	y_{112}	56.66	13.13	30.00	3.84
8	2/3	0	1/3	y_{113}	56.66	0.00	43.33	4.24
9	0	2/3	1/3	y_{223}	30.00	26.66	43.33	5.06
10	1/3	1/3	1/3	y_{123}	43.33	13.33	43.33	4.27
11*	1/2	1/4	1/4	y_{1123}	50.00	10.00	40.00	4.09
12*	1/4	1/4	1/2	y_{1223}	40.00	10.00	50.00	3.75
13*	3/4	1/4	0	y_{1112}	60.00	10.00	30.00	3.83
14*	3/4	0	1/4	y_{1113}	60.00	0.00	40.00	4.04
15*	1/4	3/4	0	y_{1222}	40.00	30.00	30.00	4.56

* Control points

The design matrix has been defined for a third-order regression model, as previous research proved that such a model may adequately describe experimental outcomes. Regression coefficients are determined from the relations:

$$\beta_i = y_i \Rightarrow \beta_1 = y_1 = 3.92; \beta_2 = y_2 = 4.75; \quad \beta_3 = y_3 = 4.95;$$

$$\beta_{ij} = \frac{9}{4}\left(y_{iij} + y_{ijj} - y_i - y_j\right) \Rightarrow \beta_{12} = -1.31;\ \beta_{13} = -0.43;\ \beta_{23} = -0.4;$$

$$\gamma_{ij} = \frac{9}{4}\left(3y_{iij} - 3y_{ijj} - y_i + y_j\right) \Rightarrow \gamma_{12} = -0.90; \quad \gamma_{13} = 0.97; \quad \gamma_{23} = 2.07;$$

$$\beta_{ijk} = 27y_{ijk} - \frac{27}{4}\left(y_{iij} + y_{ijj} + y_{ijk} + y_{ikk} + y_{jjk} + y_{jkk}\right) + \frac{9}{2}\left(y_i + y_j + y_k\right);$$

$$\beta_{123} = -3.31.$$

The regression model has the form:

$$\begin{aligned}\widehat{y} = {} & 3.92X_1 + 4.75X_2 + 4.95X_3 - 1.31X_1X_2 - 0.43X_1X_3 - 0.4X_2X_3 \\ & -0.90X_1X_2(X_1 - X_2) + 0.97X_1X_3(X_1 - X_3) + 2.07X_2X_3(X_2 - X_3) \\ & -3.31X_1X_2X_3\end{aligned} \tag{3.99}$$

It should be noted that pseudocomponents or coded factors appear in the regression model. A check of lack of fit of the regression model in control points has shown that the regression model is adequate with 95 % confidence.

Example 3.17 [18]

A researcher's objective is to the establish optimal composition of a composite rocket propellant by ballistic properties such as burning rate and specific impulse. To achieve this, an extreme vertices design of experiment has been set up for these three components of the composition:

x_1-ammonium perchlorate	65–77 %
x_2-aluminum powder	8–20 %
x_3-polyurethane binder	15–27 %

The geometric interpretation of the local factor space is given in Fig. 3.17.

Relations between the coded and real ratios are given as follows:

$$\begin{cases} x_1 = 0.77X_1 + 0.65X_2 + 0.65X_3 \\ x_2 = 0.08X_1 + 0.20X_2 + 0.08X_3 \\ x_3 = 0.15X_1 + 0.15X_2 + 0.27X_3 \end{cases} \tag{3.100}$$

The extreme vertices design for a third-order regression model is given in Table 3.35. Regression coefficients for the impulse and burning rate at pressure of 70 bar and temperature of 25 °C are determined from experimental outcomes.

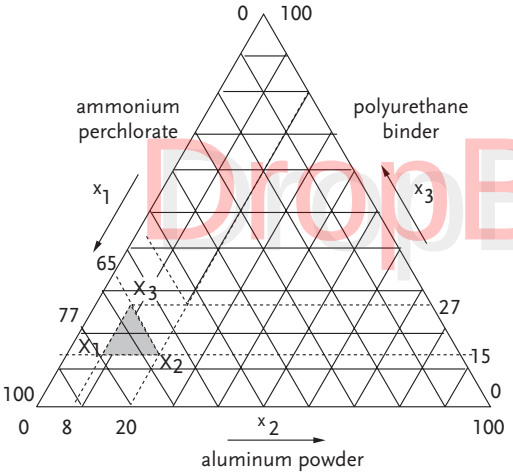

Figure 3.17 Local factor space

Table 3.35 Extreme vertices design

No.	Design matrix			Response marks	Operational matrix			Measur. response		Pred. Response	
	X_1	X_2	X_3		x_1	x_2	x_3	Y_1	Y_2	$\widehat{y}_1$	$\widehat{y}_2$
1	1	0	0	y_1	77.0	8.0	15.0	2275.1	1.40	2275.10	1.400
2	0	1	0	y_2	65.0	20.0	15.0	2200.0	1.11	2200.00	1.110
3	0	0	1	y_3	65.0	8.0	27.0	1853.5	0.54	1853.50	0.540

Table 3.35 (continued)

No.	Design matrix			Response marks	Operational matrix			Measur. response		Pred. Response	
	X_1	X_2	X_3		x_1	x_2	x_3	Y_1	Y_2	$\widehat{y}_1$	$\widehat{y}_2$
4	1/3	2/3	0	y_{122}	69.0	16.0	15.0	2255.5	1.15	2232.57	1.139
5	1/3	0	2/3	y_{133}	69.0	8.0	23.0	2030.0	0.75	2009.13	0.742
6	0	1/3	2/3	y_{233}	65.0	12.0	23.0	1961.3	0.67	1941.87	0.663
7	2/3	1/3	0	y_{112}	73.0	12.0	15.0	2265.3	1,,24	2242.57	1.229
8	2/3	0	1/3	y_{113}	73.0	8.0	19.0	2128.0	0.90	2148.26	0.896
9	0	2/3	1/3	y_{223}	65.0	16.0	19.0	2098.6	0.62	2077.37	0.618
10	1/3	1/3	1/3	y_{123}	69.0	12.0	19.0	2255.5	0.93	2230.27	0.921
11*	1/2	1/4	1/4	y_{1123}	71.0	11.0	18.0	2118.2	1.00	2266.73	1.01
12*	1/4	1/4	1/2	y_{1233}	68.0	11.0	21.0	1980.9	0.66	2172.36	0.87
13*	3/4	1/4	0	y_{1112}	74.0	11.0	15.0	2196.7	1.17	2265.97	1.27
14*	3/4	0	1/4	y_{1113}	74.0	8.0	18.0	2137.8	0.93	2155.19	0.98
15*	1/4	3/4	0	y_{1222}	68.0	17.0	15.0	–	1.08	2247.70	1.14

* Control points

1. Third-order model for specific impulse

$$\beta_1 = 2275.1; \quad \beta_2 = 2200.0; \quad \beta_3 = 1853.5; \quad \beta_{12} = 102.83; \; \beta_{13} = 66.15;$$
$$\beta_{23} = 14.40; \; \gamma_{12} = -102.83; \; \gamma_{13} = -287.10; \; \gamma_{23} = 147.15; \; \beta_{123} = 3390.98.$$
$$\widehat{y} = 2275.1X_1 + 2200.0X_2 + 1853.5X_3 + 102.83X_1X_2 + 66.15X_1X_3$$
$$+14.40X_2X_3 - 102.83X_1X_2(X_1 - X_2) - 287.10X_1X_3(X_1 - X_3)$$
$$+147.15X_2X_3(X_2 - X_3) + 3390.98X_1X_2X_3 \qquad (3.101)$$

1.1 Check of lack of fit

According to preliminary information it is:

$$S^2_{\bar{y}} = \frac{1}{N}\sum_{u=1}^{N} S^2_y = 156.06; \; S^2_y = \frac{1}{n-1}\sum_{k=1}^{n}\left(y_{uk} - \bar{y}\right)^2;$$

N=2; f=N(u-1)=2(2-1)=2; α=0.05; ℓ=4; n=2.

a) for control point 11 it is:

$$\xi = \sum a_i^2 + \sum a_{ij}^2; \; a_i = X_i(2X_i - 1); \; a_{ij} = 4X_iX_j;$$

$$a_1 = 0.5(2 \times 0.5 - 1) = 0.0; \qquad a_{12} = 4 \times 0.5 \times 0.25 = 0.5;$$
$$a_2 = 0.5(2 \times 0.25 - 1) = -0.125; \qquad a_{13} = 4 \times 0.5 \times 0.25 = 0.5;$$
$$a_3 = 0.5(2 \times 0.25 - 1) = -0.125; \qquad a_{23} = 4 \times 0.25 \times 0.25 = 0.25;$$

$$\xi = 0.59; \quad \Delta y_{11} = \left|\bar{y}_{11} - \widehat{y}_{11}\right| = 2118.2 - 2266.7 = 148.5$$

$$t_{\alpha/l;f} = \frac{\Delta y_{11}\sqrt{n}}{S_{\bar{y}}\sqrt{1+\xi}} = \frac{148.5\sqrt{2}}{12.49\sqrt{1+0.59}} = 13.30 \succ t_{T(0.01;2)} = 9.92.$$

The regression model (3.101) is not adequate in point number 11.

b) for control point 15 it is:

$a_1 = 0.75(2 \times 0.75 - 1) = 0.38;$ $a_{12} = 4 \times 0.75 \times 0.0 = 0.0;$

$a_2 = 0.05(2 \times 0.0 - 1) = 0.0;$ $a_{13} = 4 \times 0.75 \times 0.25 = 0.75;$

$a_3 = 0.25(2 \times 0.25 - 1) = -0.125;$ $a_{23} = 4 \times 0.0 \times 0.25 = 0.00;$

$$\xi = 0.72; \Delta y_{15} = \left|\bar{y}_{15} - \widehat{y}_{15}\right| = 2137.8 - 2155.19 = 17.39.$$

$$t_R = \frac{17.39\sqrt{2}}{12.49\sqrt{1+0.72}} = 1.50 \prec t_{T(0.01;2)} = 9.92$$

Table 3.36 Check of lack of fit of regression – Eq. (3.101)

No.	Coded ratios			$\bar{y}$	$\widehat{y}$	$\lvert\Delta y\rvert$	ξ	t_R	t_T	adequacy
	X_1	X_2	X_3							
11	0.50	0.25	0.25	2118.2	2266.7	148.50	0.59	13.30	9.92	Inadequate
12	0.25	0.25	0.50	1980.9	2172.3	191.46	0.59	17.15	9.92	Inadequate
14	0.25	0.75	0.00	2196.7	2265.9	69.27	0.72	5.97	9.92	Adequate
15	0.75	0.00	0.25	2137.8	2155.2	17.39	0.72	1.50	9.92	Adequate

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The regression model is adequate. A check of lack of fit of regression model (3.101) in all control points is given in Table 3.36.

2. Third-order model for burning rate

$\beta_1 = 1.40;$ $\beta_2 = 1.11;$ $\beta_3 = 0.54;$ $\beta_{12} = -0.27;$ $\beta_{13} = -0.65;$

$\beta_{23} = -0.81;$ $\beta_{123} = 2.86;$ $\gamma_{12} = -0.045; \gamma_{13} = -0.923; \gamma_{23} = -1.62.$

$$\widehat{y} = 1.40X_1 + 1.11X_2 + 0.54X_3 - 0.27X_1X_2 - 0.65X_1X_3 - 0.81X_2X_3$$
$$-0.045X_1X_2(X_1 - X_2) - 0.923X_1X_3(X_1 - X_3) - 1.62X_2X_3(X_2 - X_3) + 2.86X_1X_2X_3 \quad (3.102)$$

A check of lack of fit of the obtained regression model is given in Table 3.37.

Table 3.37 Check of lack of fit of regression – Eq. (3.102)

No.	Coded ratios			$\bar{y}$	$\hat{y}$	$\lvert\Delta y\rvert$	ξ	t_R	t_T	adequacy
	X_1	X_2	X_3							
11	0.50	0.25	0.25	1.00	1.01	0.01	0.59	0.165	9.92	Adequate
12	0.25	0.25	0.50	0.66	0.87	0.21	0.59	3.45	9.92	Adequate
13	0.25	0.75	0.00	1.08	1.14	0.06	0.70	0.950	9.92	Adequate
14	0.75	0.25	0.00	1.17	1.27	0.10	0.72	1.580	9.92	Adequate
15	0.75	0.00	0.25	0.93	0.98	0.05	0.72	0.790	9.92	Adequate

The geometric interpretation in the form of contour graphs for both regression models is given in Figs. 3.18 and 3.19.

By overlapping the simplex with specific impulse and burning rate contour lines, we can determine the optimal composition of a composite rocket propellant in a very simple way.

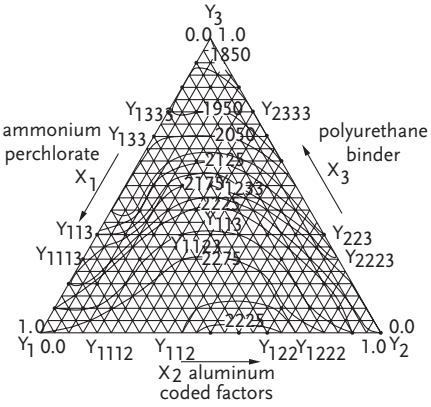

Figure 3.18 Extreme vertices design of impulse contour lines Ns/kg

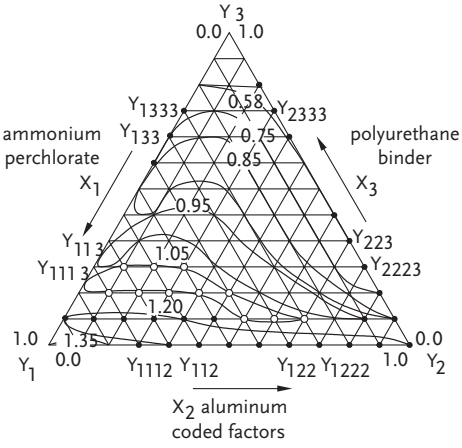

Figure 3.19 Extreme vertices design of burning rate contour lines cm/s

3.6 D-optimal Designs

The most important among the known criteria of design optimality is the requirement of *D*- and *G*-optimality. A design is said to be *D*-optimal when it minimizes the volume of the scatter ellipsoid for estimates of regression equation coefficients. The property of *G*-optimality provides the least maximum variance of predicted response values in a region under investigation.

Simplex-lattice designs exhibit the properties of *D*- and *G*-optimality in the building of second- and incomplete third-degree polynomials only. *Scheffe's* designs of higher degree are not *D*-optimal [19]. A D-optimal simplex lattice for the third-degree polynomial was deduced later by *Kiefer* [20]. If we consider a set of designs with the coordinates of points:

$$\begin{aligned} &X_i = 1;\ X_j = X_k = 0 \\ &X_i = 1 - X_j = b;\quad X_k = 0;\quad b \prec 1/2 \\ &X_i = X_j = X_k = 1/3 \end{aligned} \tag{3.103}$$

then to produce a third-degree polynomial a design will be *D*-optimal at: $b = (1 - \sqrt{5})/2$.

In the example the points in the faces of simplex are taken with coordinates: X_f=0.2764 and X_f=0.7236 [12].

Table 3.38 tabulates the *D*-optimal design for the derivation of a ternary system third-degree polynomial. Following this design the coefficients are obtained for a third-degree polynomial having the same form as that from a conventional simplex lattice:

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Table 3.38 D-optimal design for a ternary system third-degree polynomial {3,3}

N	X_1	X_2	X_3	y	N	X_1	X_2	X_3	Y
1	1	0	0	y_1	**6**	0.7236	0	0.2764	y_{113}
2	0	1	0	y_2	**7**	0.2764	0	0.7236	y_{133}
3	0	0	1	y_3	**8**	0	0.7236	0.2764	y_{223}
4	0.7236	0.2764	0	y_{112}	**9**	0	0.2764	0.7236	y_{233}
5	0.2764	0.7236	0	y_{122}	**10**	0.333	0.333	0.333	y_{123}

$$\widehat{y} = \sum_{1\le i\le q} \beta_i X_i + \sum_{1\le i\prec j\le q} \beta_{ij} X_i X_j + \sum_{1\le i\prec j\le q} \gamma_{ij} X_i X_j \left(X_i - X_j\right) + \sum_{1\le i\prec j\prec k\le q} \beta_{ijk} X_i X_j X_k \tag{3.104}$$

Formulas for the polynomial coefficients are derived by substituting the coordinates of points into the regression equation:

$$\begin{aligned}
\beta_i &= \gamma_i \\
\beta_{ij} &= \frac{5}{2}\left(\gamma_{iij} + \gamma_{ijj} + \gamma_i - \gamma_j\right) \\
\gamma_{ij} &= \frac{5}{2}\left[5\left(\gamma_{iij} - \gamma_{ijj}\right) - \gamma_i + \gamma_j\right] \\
\beta_{ijk} &= 27\gamma_{ijk} - \frac{15}{2}\left(\gamma_{iij} + \gamma_{ijj} + \gamma_{iik} + \gamma_{jkk} + \gamma_{jjk}\right) + 6\left(\gamma_i + \gamma_j + \gamma_k\right)
\end{aligned} \tag{3.105}$$

The adequacy test and the assignment of confidence intervals using a *D*-optimal design (Table 3.38) are accomplished along the same lines, as in the simplex-lattice method. The variation of ξ with composition, are given in the reference literature [12]. In constructing the fourth-order polynomial for the ternary system, the design will be *D*-optimal at:

$$X_i = \left(7 - \sqrt{21}\right)/14; X_j = 1 - X_i; X_k = 0 \tag{3.106}$$

or

X_i=0.1727; X_j=0.8273; X_k=0

Moreover, in the fourth-order *D*-optimal design there are points with coordinates:

$$X_i = X_j = \left(7 - \sqrt{5}\right)/22; X_k = 1 - \left(X_i + X_j\right) \tag{3.107}$$

or

X_i=X_j=0.2165; X_k=0.5670

In Table 3.39 a fourth-order *D*-optimal design for a ternary system is presented.

Table 3.39 D-optimal design for a ternary system fourth-degree polynomial {3,4}

N	X_1	X_2	X_3	y	N	X_1	X_2	X_3	Y
1	1	0	0	y_1	**9**	0.8273	0	0.1727	y_{1113}
2	0	1	0	y_2	**10**	0.1727	0	0.8273	y_{1333}
3	0	0	1	y_3	**11**	0	0.8273	0.1727	y_{2223}
4	0.5	0.5	0	y_{12}	**12**	0	0.1727	0.8273	y_{2333}
5	0.5	0	0.5	y_{13}	**13**	0.5670	0.2165	0.2165	y_{1123}
6	0	0.5	0.5	y_{23}	**14**	0.2165	0.5670	0.2165	y_{1223}
7	0.8273	0.1727	0	y_{1112}	**15**	0.2165	0.2165	0.5670	y_{1233}
8	0.1727	0.8273	0	y_{1222}					

According to this design the coefficients are obtained for a regression equation of the form:

$$\begin{aligned}
\widehat{y} = {} & \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \gamma_{12} X_1 X_2 (X_1 - X_2) \\
& + \gamma_{13} X_1 X_3 (X_1 - X_3) + \gamma_{23} X_2 X_3 (X_2 - X_3) + \delta_{12} X_1 X_2 (X_1 - X_2)^2 \\
& + \delta_{13} X_1 X_3 (X_1 - X_3)^2 \\
& + \delta_{23} X_2 X_3 (X_2 - X_3)^2 + \beta_{1123} X_1^2 X_2 X_3 + \beta_{1223} X_1 X_2^2 X_3 + \beta_{1233} X_1 X_2 X_3^2
\end{aligned} \tag{3.108}$$

Substituting the coordinates of points into the regression equation gives the relationships for calculation of the coefficients of the fourth-degree polynomial:

$$b_i = y_i \tag{3.109}$$

$$b_{ij} = 4y_{ij} - 2y_i - 2y_j \tag{3.110}$$

$$\gamma_{ij} = \frac{7}{6}\left[3\left(-y_i + y_j\right) + \sqrt{21}\left(y_{iiij} - y_{ijjj}\right)\right] \tag{3.111}$$

$$\delta_{ij} = \frac{7}{6}\left[-3\left(y_i + y_j\right) - 8y_{ij} + 7\left(y_{iiij} + y_{ijjj}\right)\right] \tag{3.112}$$

$$\begin{aligned}\beta_{ijk} = {} & 26.657y_i - 6.167\left(y_j + y_k\right) - 16.96\left(y_{ij} + y_{ik}\right) + 0.511y_{ik} \\ & -32.18\left(y_{iiij} + y_{iiik}\right) + 17.196\left(y_{ijjj} + y_{ikkk}\right) + 5.72\left(y_{jjjk} + y_{jkkk}\right) \\ & +84.11y_{iijk} - 23.237\left(y_{ijjk} + y_{ijkk}\right)\end{aligned} \tag{3.113}$$

$i \neq j \neq k$; i, j, k=1, 2, 3

Figure 3.20 shows the arrangement of points in *D*-optimal designs for ternary systems.

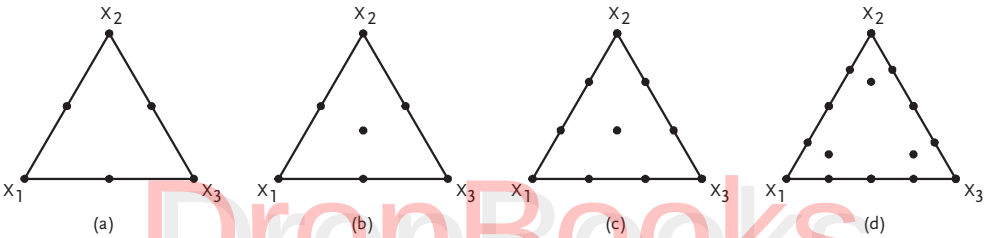

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Figure 3.20 Arrangement of points in the D-optimal designs of: a) second-order; b) incomplete third-order; c) third-order; d) fourth-order

Example 3.18 [12]

The variation of viscosity (y) of solutions in the system $(NH_4)_2HPO_4$-K_2CO_3-H_2O with composition and temperature is studied. The experimental design is conducted within a local region of the concentration triangle bounded by a saturation line at 0 °C (Figs. 3.21 and 3.22). The local region of the diagram was a triangle with the vertices: Z_1(42; 0; 58), Z_2(0; 30; 70) and Z_3(0; 0; 100).

The third-order *D*-optimal design is prepared relative to pseudocomponents Z_1, Z_2 and Z_3; and the content of initial components at the design points is determined by Eq. (3.84). Table 3.40 presents the experimental conditions both in terms of pseudocomponents and on the natural scale (per cent). The sample variance here is: S_y=0.53; and the number of degrees of freedom is f=13. From Eq. (3.105) for viscosity at 0 °C the coefficients have been calculated for the third-order regression equation:

$$\widehat{y}_1 = 8.33Z_1 + 4.99Z_2 + 1.79Z_3 - 6.95Z_1Z_2 - 9.05Z_1Z_3 - 1.37Z_2Z_3 + 17.90Z_1Z_2(Z_1 - Z_2) + 9.90Z_1Z_3(Z_1 - Z_3) + 12.37Z_2Z_3(Z_2 - Z_3) + 18.06Z_1Z_2Z_3 \quad (3.114)$$

and at 30 °C:

$$\widehat{y}_2 = 3.83Z_1 + 2.54Z_2 + 0.80Z_3 - 8.77Z_1Z_2 - 3.10Z_1Z_3 - 0.87Z_2Z_3 + 5.27Z_1Z_2(Z_1 - Z_2) + 6.55Z_1Z_3(Z_1 - Z_3) + 5.77Z_2Z_3(Z_2 - Z_3) + 3.00Z_1Z_2Z_3 \quad (3.115)$$

Table 3.40 D-optimal design {3,3}

N	Z_1	Z_2	Z_3	x_1	x_2	x_3	y_1	y_2
1	1	0	0	42.0	0	58.0	8.33	3.83
2	0	1	0	0	30.0	70.0	4.99	2.54
3	0	0	1	0	0	100.0	1.79	0.80
4	0.2764	0.7236	0	11.6	21.71	66.69	4.22	2.09
5	0.7236	0.2764	0	30.4	8.29	61.31	6.32	2.77
6	0.2764	0	0.7236	11.6	0	88.4	2.20	1.13
7	0.7236	0	0.2764	30.4	0	69.6	4.30	2.26
8	0	0.2764	0.7236	0	8.29	91.71	2.30	1.09
9	0	0.7236	0.2764	0	21.71	78.29	3.93	1.90
10	0.333	0.333	0.333	14.0	10.0	76.0	3.59	1.64
11*	0.22	0.22	0.56	9.1	6.5	84.4	2.00	1.23
12*	0.22	0.56	0.22	9.1	17.0	73.9	3.68	1.82
13*	0.56	0.22	0.22	23.9	6.5	69.6	4.70	2.12

* Control points

The results of testing the adequacy of Eqs. (3.114) and (3.115) are arrayed in Table 3.41.

Table 3.41 Check of lack of fit

N	y_1	$\widehat{y}_1$	$\lvert\Delta y_1\rvert$	y_2	$\widehat{y}_2$	$\lvert\Delta y_2\rvert$	ξ	t_{R1}	t_{R2}
11	2.0	1.68	0.32	1.23	0.72	0.51	0.8	0.77	1.22
12	3.68	3.68	0	1.82	1.82	0	0.8	0	0
13	4.70	5.70	1.00	2.12	2.59	0.47	0.9	2.33	1.09

The table value of Student's t-test is $t_{0.016;13}$=2.85. For all the test points the values of the t-ratio were found to be less than the table value, hence the regression equa-

tions Eq. (3.114) and (3.115) adequately fit the experiment with 95% level of confidence. Relations between pseudocomponents and natural variables are as follows:

$$
\begin{aligned}
1 &= 0.42Z_1^{(1)} + 0Z_1^{(2)} + 0.58Z_1^{(3)} \\
0 &= 0Z_1^{(1)} + 0.3Z_1^{(2)} + 0.7Z_1^{(3)} \\
0 &= 0Z_1^{(1)} + 0Z_1^{(2)} + 1Z_1^{(3)} \\
0 &= 0.42Z_2^{(1)} + 0Z_2^{(2)} + 0.58Z_2^{(3)} \\
1 &= 0Z_2^{(1)} + 0.3Z_2^{(2)} + 0.7Z_2^{(3)} \\
0 &= 0Z_2^{(1)} + 0Z_2^{(2)} + 1Z_2^{(3)}
\end{aligned}
\tag{3.116}
$$

The solutions to the sets of Eq. (3.116) are:

$$
\begin{aligned}
Z_1^{(1)} &= 2.38; \quad & Z_2^{(1)} &= 0; \\
Z_1^{(2)} &= 0; \quad & Z_2^{(2)} &= 3.33; \\
Z_1^{(3)} &= 0; \quad & Z_2^{(3)} &= 0
\end{aligned}
\tag{3.117}
$$

Substituting Eq. (3.117) into the set of equations (3.86), we arrive at:

$$
\begin{aligned}
Z_1 &= 2.38(1 - x_2 - x_3) \\
Z_2 &= 3.33x_2 \\
Z_3 &= 1 - Z_1 - Z_2 = 2.38x_3 - 0.95x_2 - 1.38
\end{aligned}
\tag{3.118}
$$

The geometric interpretation of the local factor space and arrangement of the points are given in Figs. 3.21 and 3.22.

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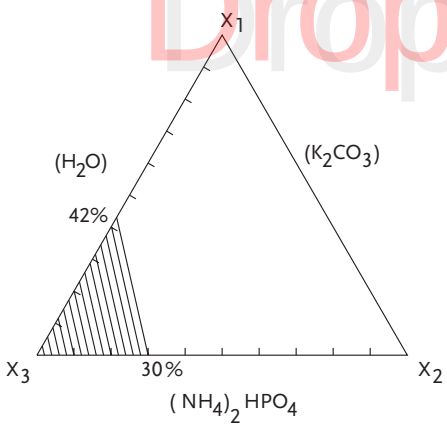

Figure 3.21 Local factor space

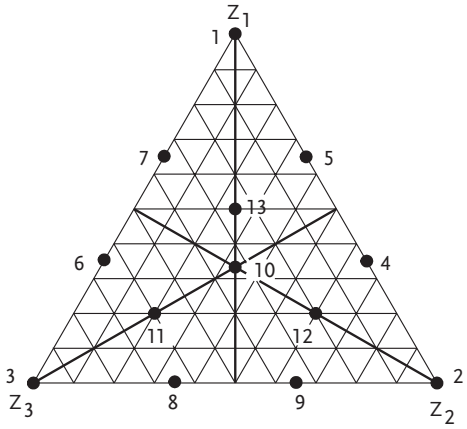

Figure 3.22 Arrangement of points

Example 3.19 [12]

We seek to determine an optimal composition of a multicomponent solvent utilized to remove hydrocarbons from yeast. The major index of purification here is the hydrocarbon content in biomass upon extraction (y). For technological and economic reasons, the experimental design is accomplished in a local section of the concentration, Fig. 3.23.

In the region covered, the mixture contains, in per cent: acetones, $X_1 \leq 74$; hexane, $X_2 \leq 90$; and water, $X_3 \leq 10$. The local portion of the diagram is a triangle with the vertices: Z_1 (9.5; 89.5; 1); Z_2 (58.5; 40; 1.5); Z_3 (74; 16; 10).

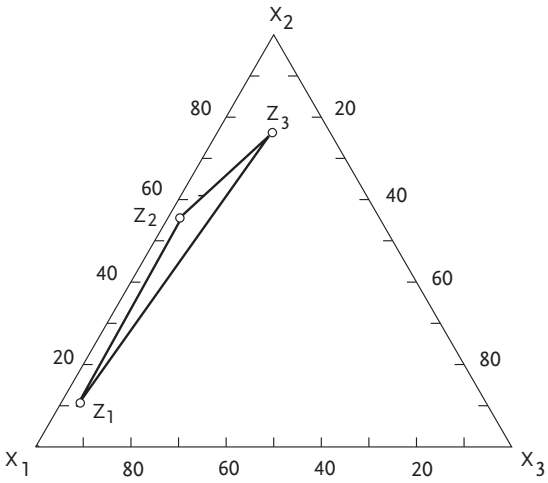

Figure 3.23 Local factor space

A fourth-order *D*-optimal design is produced with reference to pseudocomponents Z_1;Z_2 and Z_3 – Table 3.42. The pseudocomponents satisfy the principal condition for *Scheffe's* designs. The conversion to initial components at any point within the local simplex studied is carried out from Eq. (3.84). According to this design, an experiment is run with mixtures, each observation being repeated twice. Using Eqs. (3.109)–(3.113) the coefficients of fourth-order regression equation are calculated in pseudocomponents

$$\begin{aligned}\widehat{y}_u = {} & 0.1Z_1 + 0.3Z_2 + 0.04Z_3 - 0.48Z_1Z_2 - 0.04Z_1Z_3 - 0.44Z_2Z_3 \\ & +0.914Z_1Z_2(Z_1 - Z_2) - 0.312Z_1Z_3(Z_1 - Z_3) - 1.39Z_2Z_3(Z_2 - Z_3) \\ & -1.003Z_1Z_2(Z_1 - Z_2)^2 + 0.747Z_1Z_3(Z_1 - Z_3)^2 + 0.782Z_2Z_3(Z_2 - Z_3)^2 \\ & +1.398Z_1^2Z_2Z_3 + 8.416Z_1Z_2^2Z_3 - 4.703Z_1Z_2Z_3^2 \end{aligned} \quad (3.119)$$

Table 3.42 D-optimal design {3.4}

N	Z_1	Z_2	Z_3	x_1	x_2	x_3	y
1	1	0	0	9.5	89.5	1	0.1
2	0	1	0	58.5	40.0	1.5	0.3
3	0	0	1	74.0	16.0	10.0	0.04
4	0.5	0.5	0	34.0	64.7	1.3	0.08
5	0.5	0	0.5	41.7	52.8	5.5	0.06
6	0	0.5	0.5	66.2	28.2	5.8	0.06
7	0.176	0.824	0	49.9	48.7	1.4	0.05
8	0.824	0.176	0	18.12	80.79	1.09	0.09
9	0.176	0	0.824	62.6	29.0	8.4	0.12
10	0.824	0	0.176	20.85	76.55	2.6	0.1
11	0	0.176	0.824	71.22	20.30	8.49	0.2
12	0	0.824	0.176	61.25	35.75	3.0	0.11
13	0.216	0.216	0.568	56.7	37.12	6.18	0.11
14	0.216	0.568	0.216	51.2	45.65	3.15	0.091
15	0.568	0.216	0.216	34.0	62.97	3.03	0.11
16	0.333	0.333	0.333	47.3	48.5	4.2	0.108

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The sets of Eq. (3.87) under the constraints on the component content in the solvent have the form:

$$\left.\begin{aligned} 9.5Z_1^{(1)} + 89.5Z_1^{(2)} + 1Z_1^{(3)} &= 1 \\ 58.5Z_1^{(1)} + 40Z_1^{(2)} + 1.5Z_1^{(3)} &= 0 \\ 74Z_1^{(1)} + 16Z_1^{(2)} + 10Z_1^{(3)} &= 0 \end{aligned}\right\}$$
$$\left.\begin{aligned} 9.5Z_2^{(1)} + 89.5Z_2^{(2)} + 1Z_2^{(3)} &= 0 \\ 58.5Z_2^{(1)} + 40Z_2^{(2)} + 1.5Z_2^{(3)} &= 1 \\ 74Z_2^{(1)} + 16Z_2^{(2)} + 10Z_2^{(3)} &= 1 \end{aligned}\right\} \quad (3.120)$$

The solutions are:

$$Z_1^{(1)} = 0.0092; \qquad Z_2^{(1)} = 0.0215;$$
$$Z_1^{(2)} = 0.0116; \qquad Z_2^{(2)} = 0.00051;$$
$$Z_1^{(3)} = 0.0495; \qquad Z_2^{(3)} = -0.1583.$$

Using the solutions found we obtain the formulas relating natural coordinates x to Z:

$$\left.\begin{aligned} Z_1 &= -0.92 + 0.0208x_2 + 0.058x_3 \\ Z_2 &= 2.15 - 0.022x_2 - 0.18x_3 \\ Z_3 &= 1 - Z_1 - Z_2 = -0.23 + 0.001x_2 + 0.121x_3 \end{aligned}\right\} \tag{3.121}$$

The adequacy of the regression equation obtained was tested by the Student's test at five test points, the results being given in Table 3.43.

Table 3.43 Check of lack of fit

x_1	x_2	x_3	y	$\widehat{y}$	t_R	t_T
47.3	48.5	4.2	0.1022	0.108	0.365	2.83
53.0	44.0	3.0	0.079	0.072	0.392	2.83
19.0	79.0	2.0	0.1	0.07	1.778	2.83
37.9	58.7	3.4	0.13	0.12	0.547	2.83
44.5	54.0	1.5	0.04	0.05	0.57	2.83

It is seen that Eq. (3.119) adequately fits the experiment at the significance level α=0.05%. The quality of the resultant product is considered satisfactory, if the content of residual hydrocarbons in the biomass is under 0.05%. With the aim to elucidate the solvent compositions meeting this requirement, the lines of constant response are plotted to Eq. (3.119); the curves are shown in Fig. 3.24. The solvent compositions meeting the requirement that y<0.05%, can be found within the area of the simplex.

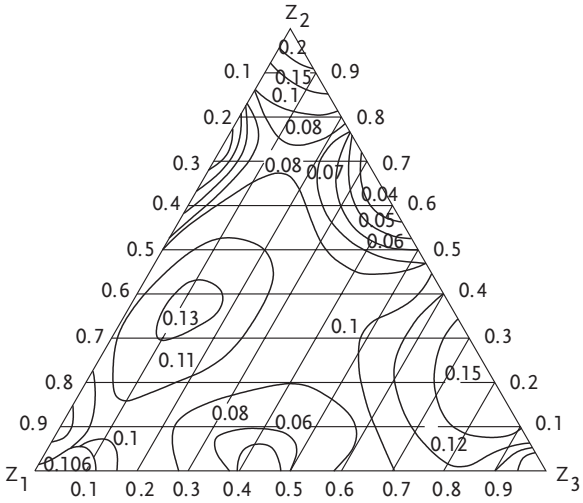

Figure 3.24 Isolines of residual hydrocarbon content of biomass

3.7
Draper-Lawrence Design

Draper and *Lawrence* [21] have proposed designs in which, unlike the simplex lattices, all the points are located within the investigation area, i.e. experiments are conducted with the q-component mixtures only. These designs make allowance for the absence of prior information on the response surface and that it is desirable to approximate an unknown response surface by low-degree polynomials. Data points are chosen to provide the best representation of a complex surface by simple polynomials. In building a polynomial of degree n_1, design points are to be selected so that a minimal systematic error results, which occurs due to the higher degree of the response function polynomial, n_2 compared with the degree of estimating the polynomial, n_1. The principles underlying the selection of suitable designs have been put forward earlier by *Box* and *Draper*. *Draper* and *Lawrence* built designs for ternary and quaternary systems and polynomials of degrees n_1=1; n_2=2; n_1=2 and n_2=3. To make the generation of design more convenient these authors introduce a new reference system. With ternary mixtures, the new coordinate system is selected in the plane of the concentration triangle (X_1, X_2, X_3), so that the origin coincides with the centroid of the triangle, one of the triangle axes lies on the axis Z_2 and the two others are symmetrical about this axis, Fig. 3.25.

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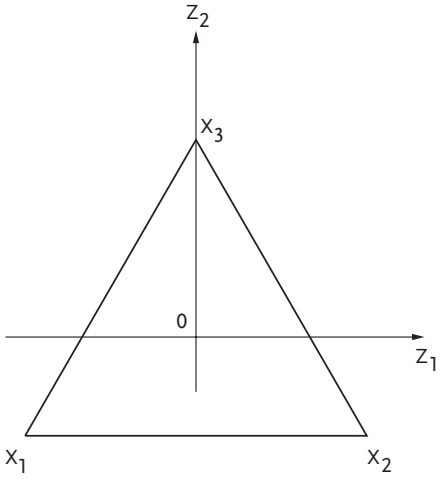

Figure 3.25 Coordinate system for the designs of Draper and Lawrence

The triangular coordinate system (X_1, X_2, X_3) is related to the rectangular one (Z_1, Z_2) as follows:

$$\left.\begin{aligned} Z_1 &= \frac{1}{2}(-X_1 + X_2) \\ Z_2 &= \frac{\sqrt{3}}{6}(-X_1 - X_2 + 2X_3) \end{aligned}\right\} \tag{3.122}$$

$$\left.\begin{aligned} X_1 &= \frac{1}{3}\left(-3Z_1 - Z_2\sqrt{3} + m\right) \\ X_2 &= \frac{1}{3}\left(3Z_1 - Z_2\sqrt{3} + m\right) \\ X_3 &= \frac{1}{3}\left(2Z_2\sqrt{3} + m\right) \end{aligned}\right\} \tag{3.123}$$

where:
m-is the length of a side of the concentration triangle.

When the whole of the diagram is explored, m=1; and when local areas of the diagram are explored, m<1. Design points for ternary system are selected (in the coordinates Z_1, Z_2) from the following sets:

1) Vertices of a triangle similar to a given concentration triangle and centered on the origin, with side of length p:

$$\left[0, +\frac{1}{\sqrt{3}}p\right]\left[+\frac{1}{2}p, -\frac{\sqrt{3}}{6}p\right]\left[-\frac{1}{2}p, -\frac{\sqrt{3}}{6}p\right]$$

2) Vertices of a triangle similar to a given concentration triangle and centered on the origin, with side of length q:

$$\left[0, -\frac{1}{\sqrt{3}}q\right]\left[+\frac{1}{2}q, \frac{\sqrt{3}}{6}q\right]\left[-\frac{1}{2}q, \frac{\sqrt{3}}{6}q\right]$$

3) Vertices of a square centered on the origin, with sides 2a parallel to axes (±a, ±a);
4) Points on coordinate axes (±b, 0), (0, ±b);
5) Vertices of a rectangle (c, d), (-c, -d), (c, -d) and (-c, d).

After one or the other design of *Draper-Lawrence* has been constructed for a ternary system, first-degree polynomials are derived in two independent variables Z_1 and Z_2 (n_1=1 for n_2=2).

$$\widehat{y} = b_0 + b_1 Z_1 + b_2 Z_2 \tag{3.124}$$

or second-order polynomials (n_1=2 for n_2=3):

$$\widehat{y} = b_0 + b_1 Z_1 + b_2 Z_2 + b_{12} Z_1 Z_2 + b_{11} Z_1^2 + b_{22} Z_2^2 \tag{3.125}$$

Draper and *Lawrence* suggested that for the first-order polynomials and ternary systems (q=3), the designs of experiments should containin from 6 to 9 point-trials. Parameters for some of *Draper-Lawrence* designs (in fractions of m) at q=3, n_1=1 and n_2=2, are given in Table 3.44. If the number of design points is more than that of a selected set, then an appropriate number of points is added at the center of triangle (with coordinates Z_1=0, Z_2=0). For example, we consider a *Draper-Lawrence* design (1,2) containing six points, Table 3.45. Points of set 1 at m=1 have the coordinates (Z_1, Z_2):

$$\left[0.0; \frac{0.621}{\sqrt{3}}\right]\left[+\frac{0.621}{2}; -\frac{0.621\sqrt{3}}{6}\right]\left[-\frac{0.621}{2}; \frac{0.621\sqrt{3}}{6}\right]$$

or:

(0.0; 0.366); (0.3105; -0.18); (-0.3105; -0.18)

Points of a set 2 have the coordinates:

$$\left[0.0; -\frac{0.339}{\sqrt{3}}\right]\left[+\frac{1}{2}0.339; \frac{0.339\sqrt{3}}{6}\right]\left[-\frac{1}{2}0.339; \frac{0.339\sqrt{3}}{6}\right]$$

or:

(0.0; -0.196); (0.170; 0.098); (-0.170; 0.098)

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Table 3.44 Parameters of Draper-Lawrence designs for q=3, $n_1 = 1, n_2 = 2$

Set (n_1, n_2)	Center points	Design points-total N	Parameters	
(1,2)	0	6	p=0.621	q=0.339
(1,2)	1	7	p=0.662	q=0.381
(1,2)	2	8	p=0.699	q=0.421
(1,2)	3	9	p=0.733	q=0.457
(1,3)	0	7	p=0.616	q=0.160
(1,4)	0	7	p=0.616	b=0.226
(1,5)	0	7	p=0.616	$c = \sqrt{0.051m^2 - d^{2*}}$
(1,1,2)	0	9	p_1=0.606; q=0.364	p_2=0.500
(1,2,2)	0	9	p=0.727; q_2=0.200	q_1=0.425

* Value of d^2 is selected at random

Table 3.45 Draper-Lawrence design matrix (1,2); for q=3, $n_1 = 1, n_2 = 2$

N	Z_1	Z_2	X_1	X_2	X_3
1	0.0	0.366	0.12	0.12	0.76
2	0.311	-0.18	0.127	0.748	0.125
3	-0.311	-0.18	0.748	0.127	0.125
4	0.0	-0.196	0.447	0.447	0.106
5	0.170	0.098	0.106	0.447	0.447
6	-0.170	0.098	0.447	0.106	0.447

We make a transition from point coordinates in the system (Z_1, Z_2) to those in the triangle X_1, X_2, X_3 using Eqs. (3.123). Let us map, for example, the first point with coordinates Z_1=0.0 and Z_2=0.366 (m=1). For this point:

$$X_1 = \frac{1}{3}\left(-0.366\sqrt{3} + 1\right) = 0.12$$

$$X_2 = \frac{1}{3}\left(-0.366\sqrt{3} + 1\right) = 0.12$$

$$X_3 = \frac{1}{3}\left(2 \times 0.366\sqrt{3} + 1\right) = 0.76$$

To test the calculation we sum up:

$X_1+X_2+X_3$=0.12+0.12+0.76=1.0

The arrangement of the points in the concentration triangle is shown in Fig. 3.26.

Draper and *Lawrence* suggested for second-order polynomials of Eq. (3.125) as applied to ternary systems, the designs containing from 8 to 15 design points. Parameters for the *Draper-Lawrence* designs (in fractions of m) at q=3, n_1=2 and n_2=3, are summarized in Table 3.46.

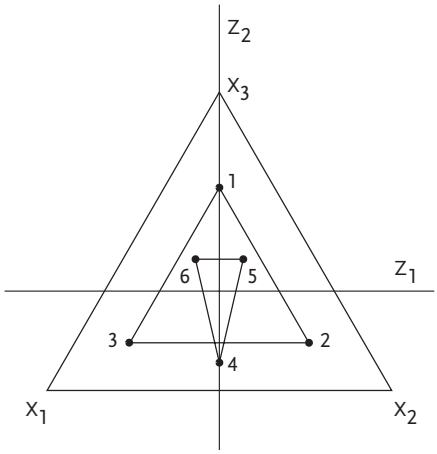

Figure 3.26 Draper-Lawrence design (1,2)

Table 3.46 Parameters of Draper-Lawrence designs at q=3, $n_1 = 2, n_2 = 3$

n_1, n_2, n_1, n_3	n_0	N	Parameters				
(1,2)	1	7	p=0.670	q=0.385			
(1,2)	2	8	p=0.698	q=0.421			
(1,2)	3	9	p=0.723	q=0.450			
(1,1,2)	0	9	p_1=0.715	p_2=0.233	q=0.430		
(1,1,2)	1	10	p_1=0.729	p_2=0.323	q=0.445		
(1,1,2)	2	11	p_1=0.738	p_2=0.398	q=0.462		
(1,1,2)	3	12	p_1=0.743	p_2=0.465	q=0.450		
(1,1,2)	4	13	p_1=0.742	p_2=0.532	q=0.485		
(1,2,2)	0	9	p=0.716	q_1=0.342	q_2=0.342		
(1,2,2)	1	10	p=0.739	q_1=0.367	q_2=0.367		
(1,1,1,2)	0	12	p_1=0.751	p_2=0.422	p_3=0.189	q=0.470	
(1,1,2,2)	0	12	p_1=0.748	p_2=0.445	q_1=0.468	q_2=0.156	
(1,2,2,2)	0	12	p=0.782	q_1=0.348	q_2=0.348	q_3=0.348	
(1,3,4)	2	13	p=0.756	a=0.183	b=0.258		
(1,3,5)	2	13	p=0.756	a=0.300	c=0.547	d=0.130	
(1,4,5)	2	13	p=0.756	b=0.212	c=0.130	d=0.257	
(1,5,5)	2	13	p=0.756	c_1=0.094	d_1=0.272	c_2=0.172	d_2=0.125
(1,1,2,5)	0	13	p_1=0.297	p_2=0.756	q=0.295	c=0.111	d=0.268
(1,1,2,5)	0	13	p_1=0.478	p_2=0.756	q=0.477	c=0.045	d=0.109
(1,1,2,5)	1	14	p_1=0.369	p_2=0.766	q=0.319	c=0.112	d=0.270
(1,1,2,5)	1	14	p_1=0.514	p_2=0.762	q=0.481	c=0.058	d=0.140
(1,1,2,5)	2	15	p_1=0.545	p_2=0.766	q=0.480	c=0.071	d=0.171

n_0-Center points

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Consider then a design (1,3,4) containing 13 points: 11 points of the sets (1,3,4) and 2 augmenting points at the center of the triangle, Fig. 3.27.

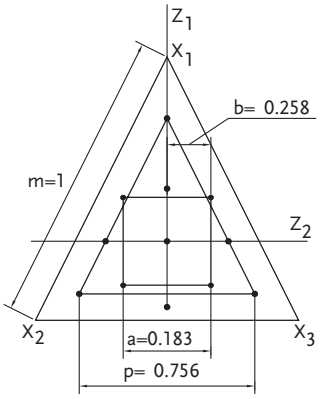

Figure 3.27 Draper-Lawrence design (1,3,4)

The points of set 1 at m=1 have the coordinates (Z_1, Z_2):

$$\left[0; \frac{0.756}{\sqrt{3}}\right]\left[+\frac{0.756}{2}; -\frac{0.756\sqrt{3}}{6}\right]\left[-\frac{0.756}{2}; \frac{0.756\sqrt{3}}{6}\right]$$

or:

(0.0; 0.437); (0.378; -0.218); (-0.378; -0.218)

The points of set 3:

(0.183; 0.183); (0.183; -0.183); (-0.183; 0.183); (-0.183; -0.183)

The points of set 4:

(0.258; 0); (-0.258; 0); (0; 0.258); (0.0; -0.258)

The experimental design is tabulated in Table 3.47.

The coordinates X_1, X_2, X_3 are related to Z_1, Z_2 by Eqs. (3.123). The coefficients of the second-order regression equation $\hat{\gamma}=f(Z_1, Z_2)$ are derived by the method of least squares. For the adequacy of fit, t-tests are applied to the experimental data at test points. The equation is adequate if the t_R-test values for all the test points are less than the table value. The t_R-test values are to be found from Eq. (3.59). Values of ξ can be taken from appropriate contour charts. Using the designs of *Draper-Lawrence,* the composition dependence of ξ can only be calculated with a digital computer. To develop designs of quaternary systems, *Draper-Lawrence* also introduced a coordinate system (Z_1, Z_2, Z_3). The origin of the new system coincides with the centroid of the concentration tetrahedron (X_1, X_2, X_3, X_4), and the coordinate axes are directed so that the four vertices of the tetrahedron in the new coordinate system form a half-replica of the full factorial design 2^3 with the defining contrast $1=Z_1Z_2Z_3$. In the new system (Z_1, Z_2, Z_3) the coordinates of the tetrahedron vertices are in the general case:

(m, m, -m); (m, -m, m); (-m, m, m); (-m, -m, -m)

and for the tetrahedron edge m=1:

(1, 1, -1); (1, -1, 1); (-1, 1, 1); (-1, -1, -1)

Table 3.47 Design matrix (1,3,4); q=3; $n_1 = 2, n_2 = 3, N = 13$

N	Z_1	Z_2	X_1	X_2	X_3
1	0.0	0.437	0.081	0.081	0.838
2	0.378	-0.218	0.081	0.837	0.082
3	-0.378	-0.218	0.837	0.081	0.082
4	0.183	0.183	0.044	0.410	0.546
5	0.183	-0.183	0.256	0.622	0.122
6	-0.183	0.183	0.410	0.045	0.545
7	-0.183	-0.183	0.622	0.256	0.122
8	0.258	0.0	0.076	0.591	0.333
9	-0.258	0.0	0.592	0.075	0.333
10	0.0	0.258	0.184	0.184	0.632
11	0.0	-0.258	0.482	0.482	0.036
12	0.0	0.0	0.333	0.333	0.333
13	0.0	0.0	0.333	0.333	0.333

The coordinate systems (X_1, X_2, X_3, X_4) and (Z_1, Z_2, Z_3), are related to each other as follows:

$$\begin{aligned} Z_1 &= X_1 + X_2 - X_3 - X_4 \\ Z_2 &= X_1 - X_2 + X_3 - X_4 \\ Z_3 &= -X_1 + X_2 + X_3 - X_4 \end{aligned} \tag{3.126}$$

and

$$\begin{aligned} X_1 &= \frac{1}{4}(Z_1 + Z_2 - Z_3 + m) \\ X_2 &= \frac{1}{4}(Z_1 - Z_2 + Z_3 + m) \\ X_3 &= \frac{1}{4}(-Z_1 + Z_2 + Z_3 + m) \\ X_4 &= \frac{1}{4}(-Z_1 - Z_2 - Z_3 + m) \end{aligned} \tag{3.127}$$

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Design points for quaternary systems are chosen (in coordinates Z_1, Z_2, Z_3) from the following sets

1) Vertices of a tetrahedron similar to the concentration one with the coordinates of the vertices:

   (a, a, -a); (a, -a, a); (-a, a, a); (-a, -a, -a)

2) Vertices of a tetrahedron:

   (b, b, b); (b, -b, b); (b, b, -b); (-b, -b, b)

3) Points on the axes:

   (±h, 0, 0); (0, ±h, 0); (0, 0, ±h)

4) Vertices of tetrahedrons with the coordinates:

$$(-r,-s,-t);(-r,s,t);(r,-s,t);(r,s,-t)$$
$$(-t,r,s);(t,-r,s);(t,r,-s);(-t,-r,-s)$$
$$(-s,t,r);(s,-t,r);(s,t,-r);(-s,-t,-r)$$

After one or other *Draper-Lawrence* design has been constructed for quaternary systems, polynomials are obtained in three independent variables Z_1, Z_2 and Z_3 of first degree (n_1=1, for n_2=2):

a) First-degree polynomial (n_1=1, for n_2=2)

$$\widehat{y}=b_0+b_1Z_1+b_2Z_2+b_3Z_3 \tag{3.128}$$

b) Second-order polynomial (n_1=2, for n_2=3)

$$\widehat{y} = b_0 + b_1Z_1 + b_2Z_2 + b_3Z_3 + b_{12}Z_1Z_2 + b_{13}Z_1Z_3 + b_{23}Z_2Z_3 + b_{11}Z_1^2 + b_{22}Z_2^2 + b_{33}Z_3^2 \tag{3.129}$$

Parameters (in fractions of m) for some designs of *Draper-Lawrence* containing no more than 12 points, at q=4, n_1=1, n_2=2, are provided in Table 3.48.

Table 3.48 Parameters of Draper-Lawrence design for q=4, n_1 = 1,n_2 = 2

Set (n_1, n_2)	Center points	Design points total-N	Parameters	
(1.2)	0	8	a=0.548	b=0.315
(1.2)	1	9	a=0.567	b=0.344
(1.2)	2	10	a=0.602	b=0.371
(1.2)	3	11	a=0.626	b=0.397
(1.2)	4	12	a=0.650	b=0.421
(1.3)	0	10	a=0.550	h=0.628
(1.3)	1	11	a=0.568	h=0.674
(1.3)	2	12	a=0.585	h=0.718
(4)	0	12	r=0.539, t=0.500	s=0.248
(4)	6	12	r=0.616, t=0.300	s=0.360

At N≥12, the values of parameters r, s, t and N are to be found from the set of equations:

$$r^2+s^2+t^2=N\times m^2/20$$
$$r\times s\times t=N\times m^3/180 \tag{3.130}$$

where:
m-is the edge of the concentration tetrahedron.

The design parameters (in fractions of m) for quaternary mixtures with n_1=2, n_3=3, and 18≤N≤24, are presented in Table 3.49.

All the above designs are built assuming that there only exists a systematic bias. In actual practice, however, besides the systematic error, the experimental data also contain random errors.

When minimizing the total error [21] the basic configuration of designs [20] may be retained with the coordinates of design points multiplied by a quantity θ>1, i.e.

for a ternary system the points (θZ_1, θZ_2), and for a quaternary one with (θZ_1, θZ_2, θZ_3) should be used. The parameter θ depends of the random error and the polynomial coefficients, being close to unity if the random error is predominant. As for each particular experiment the exact value of θ is rather difficult to find, it may, to a rather rough approximation, be taken to be equal to 1.1 for ternary and 1.2 for quaternary systems. As an example, to minimize the total error we transform the design (1, 3, 4) for q=3, n_1=2 and n_2=3, given in Table 3.47.

The coordinates of the first point for θ=1.1 are to be found as follows:

$$X_1 = \frac{1}{3}\left(-3\theta Z_1 - \theta Z_2\sqrt{3} + m\right) = \frac{1}{3}\left(-1.1 \times 0.437\sqrt{3} + 1\right) = 0.056$$

$$X_2 = \frac{1}{3}\left(+3\theta Z_1 - \theta Z_2\sqrt{3} + m\right) = \frac{1}{3}\left(3 \times 1.1 \times 0.0 - 1.1 \times 0.437\sqrt{3} + 1\right) = 0.056$$

$$X_3 = \frac{1}{3}\left(2\theta Z_2\sqrt{3} + m\right) = \frac{1}{3}\left(2 \times 1.1 \times 0.437\sqrt{3} + 1\right) = 0.888$$

The complete design of the experiment is given in Table 3.50.

Table 3.49 Parameters of Draper-Lawrence design for q=4, $n_1 = 2, n_2 = 3$

Set	Center points	N	Parameters				
			a_1	a_2	r	s	t
(1,1,4)	0	20	0.673	0.0945	0.684	0.260	0.0524
(1,1,4)	1	21	0.679	0.179	0.694	0.270	0.0564
(1,1,4)	2	22	0.685	0.248	0.702	0.274	0.0532
(1,1,4)	3	23	0.690	0.315	0.708	0.268	0.0406
(1,1,4)	4	24	0.694	0.393	0.710	0.242	0.00912
			a	b	r	s	t
(1,2,4)	1	21	0.676	0.165	0.696	0.274	0.0784
(1,2,4)	2	22	0.680	0.220	0.706	0.281	0.106
(1,2,4)	3	23	0.683	0.272	0.717	0.274	0.144
(1,2,4)	4	24	0.685	0.317	0.727	0.226	0.225
			a	h	r	s	t
(1.3,4)	0	22	0.682	0.319	0.0807	0.291	0.702
(1,3,4)	1	23	0.686	0.390	0.0925	0.306	0.708
(1,3,4)	2	24	0.690	0.459	0.104	0.321	0.710
			a_1	a_2	b	h	
(1,1,2,3)	0	18	0.292	0.667	0.279	0.765	
(1,1,2,3)	1	19	0.337	0.672	0.292	0.776	
(1,1,2,3)	2	20	0.380	0.674	0.305	0.786	
(1,1,2,3)	3	21	0.420	0.676	0.318	0.795	
(1,1,2,3)	4	22	0.460	0.674	0.332	0.805	
(1,1,2,3)	5	23	0.501	0.669	0.346	0.814	
(1,1,2,3)	6	24	0.548	0.656	0.359	0.822	
			a_1	a_2	a_3	b	h
(1,1,1,2,3)	0	22	0.679	0.442	0.132	0.326	0.805
(1,1,1,2,3)	1	23	0.683	0.455	0.191	0.332	0.814
(1,1,1,2,3)	2	24	0.691	0.441	0.288	0.340	0.822

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Table 3.49 (continued)

Set	Center points	N	Parameters				
			a_1	a_2	b_1	b_2	h
(1,1,2,2,3)	0	22	0.677	0.451	0.126	0.321	0.805
(1,1,2,2,3)	1	23	0.677	0.479	0.181	0.315	0.814
(1,1,2,2,3)	2	24	0.672	0.517	0.275	0.275	0.822
(1,1,2,3,3)	0	24	0.680	0.494	0.329	0.317	0.818

Table 3.50 Design matrix {1,3,4} for q=3, n_1=2, n_2=3, θ=1,1

N	X_1	X_2	X_3	N	X_1	X_2	X_3
1	0.056	0.056	0.888	8	0.050	0.617	0.333
2	0.056	0.888	0.056	9	0.617	0.050	0.333
3	0.888	0.056	0.056	10	0.170	0.170	0.660
4	0.016	0.418	0.566	11	0.500	0.500	0.000
5	0.248	0.651	0.101	12	0.333	0.333	0.333
6	0.418	0.016	0.566	13	0.333	0.333	0.333
7	0.651	0.248	0.101				

Example 3.20 [12]

The dependence of viscosity at 30 °C on composition is studied for the liquid complex fertilizer consisting of diammonium phosphate, potash and water $(NH_4)_2HPO_4$, K_2CO_3, H_2O. For the investigation, the region of unsaturated solutions for both salts at 30 °C is selected (Fig. 3.28), the side of the concentration triangle being m=0.5.

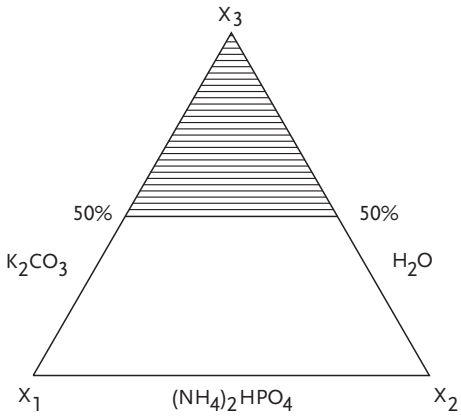

Figure 3.28 Local factor space

A *Draper-Lawrence* design containing 13 points is performed (Table 3.51). It is convenient to treat the subregion studied as a concentration triangle in the new coordinate system (X_1', X_2', X_3'):

$$X_1'+X_2'+X_3'=0.5$$

The coordinates X_j' and X_j are related by:

$$\left.\begin{aligned} X_1' &= 2X_1 \\ X_2' &= 2X_2 \\ X_3' &= 1 - X_1' - X_2' = 1 - 2X_1 - 2X_2 \end{aligned}\right\} \tag{3.131}$$

we also have, by Eq. (3.123):

$$Z_1 = \frac{1}{2}\left(-X_1' + X_2'\right) = -X_1 + X_2$$

$$Z_2 = \frac{\sqrt{3}}{6}\left(-X_1' + X_2' + 2X_3'\right) = \frac{\sqrt{3}}{3}\left(X_3 - 2X_1 - 2X_3\right) \tag{3.132}$$

The design of the experiment and measurements of viscosity for two parallel experiments are given in Table 3.51. From the table, the coefficients of the regression equation are obtained by the method of least squares,

$$\widehat{y} = 1.54 - 0.94Z_1 - 1.01Z_2 - 8.93Z_1Z_2 + 10.48Z_1^2 + 0.76Z_2^2$$

Table 3.51 Design matrix and experimental data

N	Z_1	Z_2	X_1'	X_2'	X_3'	X_1	X_2	X_3	$\widehat{y}$
1	0	0.437	0.081	0.081	0.838	0.040	0.040	0.920	1.033
2	0.378	-0.218	0.081	0.837	0.082	0.040	0.418	0.542	4.873
3	-0.378	-0.218	0.837	0.081	0.082	0.418	0.040	0.542	4.722
4	0.183	0.183	0.044	0.410	0.546	0.022	0.205	0.772	1.481
5	0.183	-0.183	0.256	0.622	0.122	0.128	0.311	0.561	3.294
6	-0.183	0.183	0.410	0.045	0.545	0.311	0.128	0.561	2.996
7	-0.183	-0.183	0.622	0.256	0.122	0.205	0.023	0.772	2.160
8	0.258	0	0.075	0.592	0.333	0.092	0.092	0.816	1.430
9	-0.258	0	0.592	0.075	0.333	0.241	0.241	0.518	3.624
10	0	0.258	0.184	0.184	0.632	0.038	0.296	0.666	2.423
11	0	-0.258	0.482	0.482	0.036	0.296	0.038	0.666	2.165
12	0	0	0.333	0.333	0.333	0.167	0.167	0.666	2.191
13	0	0	0.333	0.333	0.333	0.167	0.167	0.666	2.207

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The equation derived is found to be an adequate fit to the experiment. By Eq. (3.132) the estimated regression equation in the natural scale takes the form.

$$\widehat{y} = 1.54 + 2.1X_1 + 0.22X_2 - 0.58X_3 + 1.18X_1^2 + 21.81X_2^2 \\ -18.93X_1X_2 + 4.14X_1X_3 - 6.17X_2X_3 + 0.25X_3^2$$

Geometric interpretation of design points in Draper-Lawrence design is shown in Fig. 3.29.

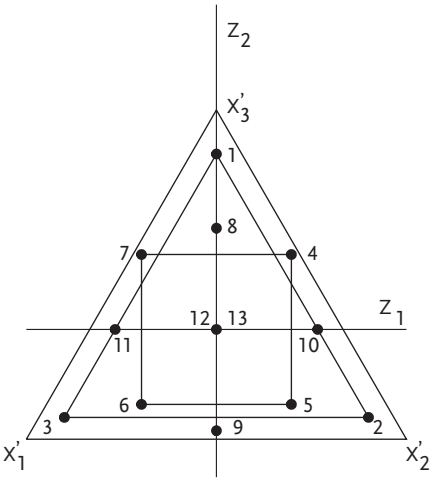

Figure 3.29 Experimental design

3.8
Factorial Experiments with Mixture

In certain practical applications it is advantageous to consider variation of properties not with absolute amounts of components, but with their ratios. If the percentage of each component is not zero, then given upper and lower constraints for the components, ratios of components may be utilized to build conventional factorial designs [22]. The number of ratios in a q-component system is q-1:

$X_1+X_2+...+X_q=1$

$$Z_1 = \frac{X_1}{X_2} \qquad Z_2 = \frac{X_3}{X_2} \ldots\ldots Z_i = \frac{X_i}{X_j} \ldots\ldots Z_{q-1} = \frac{X_{q-1}}{X_q} \tag{3.133}$$

Thus, using the component ratios as independent factors, the dimensionallity of the problem is reduced by one, and hence the number of experiments is also decreased.

Figure 3.30 shows *Kenworthy* designs [22] 2^2 and 2^3 for handling the variation of property with component ratios $Z_1=X_1/X_3$ and $Z_2=X_3/X_2$.

The points on the line originating from vertex X_2 feature a constant ratio of components X_1 and X_3. In a like manner, the line originating from vertex X_1 is the locus of equal ratios of X_3 to X_2. To meet the orthogonality condition for the design matrix the recourse is made to the linear transformation of Eq. (2.59).

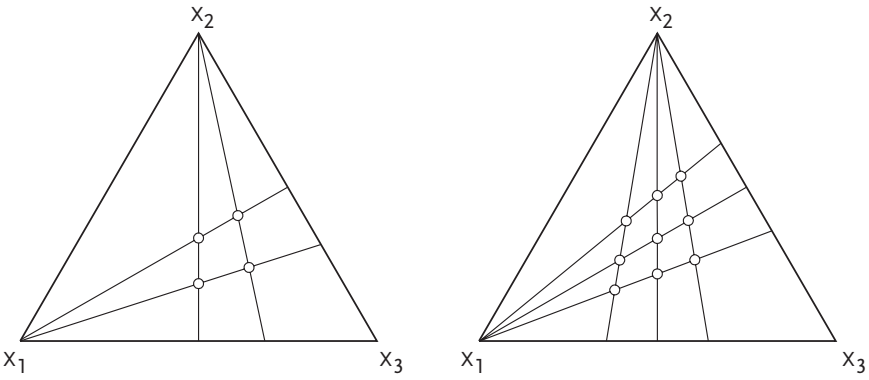

Figure 3.30 Designs using component ratios

Example 3.21

We seek the functional relationship between the yield of sodium and potassium bicarbonates and the composition of the initial sylvanite solution. The factors controlling the potassium utilization, in the carbonization process are chosen to be the per cent ratios of two of three components making up the system:

Z_1=NaCl/KCl; Z_2=H_2O/NaCl

To derive the regression equation, we shall use a second-order orthogonal design for k=2, N=9 and the star arm α=1, Fig. 3.31.

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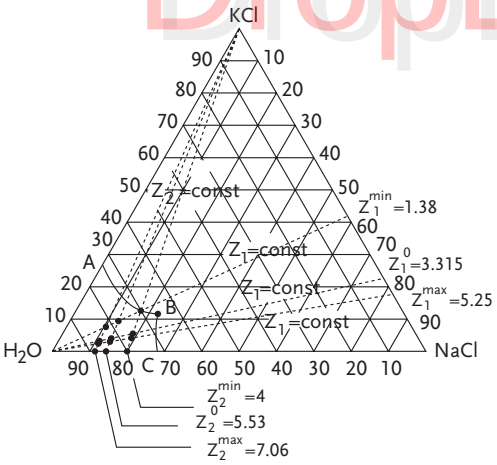

Figure 3.31 Orthogonal design of experiment

The region where independent factors will be studied is given in Table 3.52, and the design matrix in Table 3.53.

Table 3.52 Factor variation levels

Name	Z_1	Z_2
Null level	3.315	5.53
Variation interval	1.935	1.53
Upper level	5.25	7.06
Lower level	1.38	4.00

Table 3.53 Design matrix

N	X_0	X_1	X_2	X_1X_2	X_1'	X_2'	y′	y″	$\bar{y}$	S_y^2	$\widehat{y}$	$\bar{y}-\widehat{y}$	$(\bar{y}-\widehat{y})^2$
1	+	+	+	+	+1/3	+1/3	10	14	12	8	10.74	1.26	1.59
2	+	−	−	+	+1/3	+1/3	77.5	79.5	78.5	2	77.74	0.76	0.58
3	+	+	−	−	+1/3	+1/3	27	28.6	27.8	1.28	27.84	0.04	0.001
4	+	−	+	−	+1/3	+1/3	59.5	62.5	61	4.5	60.64	0.36	0.13
5	+	+	0	0	+1/3	-2/3	18.5	17.5	18	0.5	19.3	1.3	1.69
6	+	−	0	0	+1/3	-2/3	68.2	67.8	68	0.08	69.19	1.19	1.42
7	+	0	+	0	-2/3	+1/3	30	34	32	8	32.59	0.59	0.35
8	+	0	−	0	-2/3	+1/3	51	49	50	2	49.69	0.31	0.09
9	+	0	0	0	-2/3	-2/3	43	40.2	41.6	3.92	41.14	0.46	0.21

Variances of replicated design points are equal, so that the reproducibility variance is:

$$S_y^2 = \frac{\sum_{i=1}^{9} S_i^2}{9} = \frac{30.28}{9} = 3.37; f = N(n-1) = 9(2-1) = 9$$

The coefficients of the regression equation and their errors are computed from Eqs. (2.102) and (2.107).

$$b_0 = 43.21; \quad b_1 = -24.95; \quad b_2 = -8.55; \quad b_{12} = 0.425; \quad b_{11} = 3.1; \quad b_{22} = 1.1$$

$$S_{b_i} = 0.75; \quad S_{b_{ii}} = 1.3; \quad S_{b_{ij}} = 0.92$$

All regression coefficients except b_{12} and b_{22} are statistically significant with 95% confidence level and the regression equation takes the form:

$$\widehat{y} = 41.14 - 24.95X_1 - 8.55X_2 + 3.1X_1^2$$

The adequacy variance is obtained from the formula:

$$S_{AD}^2 = \frac{\sum_{i=1}^{N} \left(\bar{y}_i - \widehat{y}_i\right)^2}{N-\ell} = 2.46$$

where:

ℓ-is the number of significant coefficients.

The regression model is adequate.

3.9
Full Factorial Combined with Mixture Design-Crossed Design

In 1981, *Cornell* [23] published results of experimental studies where the quality of a fish patty was defined by both its composition and production process. The experiment included seven different mixtures prepared by mixing different species of fish and then subjecting the resulting patty to various cooking conditions and defined in accord with simplex-centroid design, Fig. 3.32. The preparation procedure of the defined mixtures or a formulation included three control process factors with associated variation levels: baking temperature from 190 °C to 218 °C; time in the oven from 25 [min] to 40 [min]; deep fat frying time from 25 s to 40 s. Process factor levels have been varied in accordance with the 2^3 full factorial experiment. Design of experiment $7{\times}2^3$ with 56 trials of a simplex-centroid design $\times$ 2^3 full factorial design has been sufficient for mathematical modelling of the observed phenomenon, Fig. 3.33. A regression model with 56 regression coefficients or reduced regression model with 18 coefficients [24] has been sufficient for an adequate description of the problem.

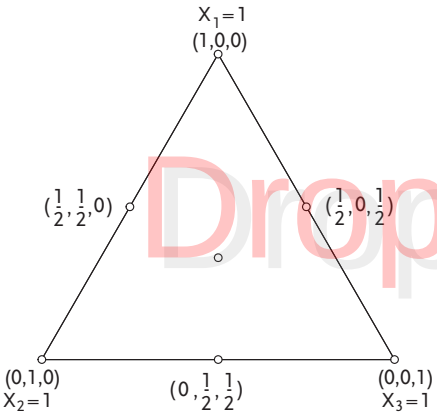

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Figure 3.32 Simplex-centroid design

This was the first example of application of a *mixture design* × *process factor design* in experimental studies. Since such designs contain a relatively large number of factors, it is of interest to replace full factorial designs of process factors with fractional factor designs. Examples of such designs with applications on an industrial level were presented in works of *Wagner* and *Gorman* [25]; *John* and *Gorman* [26]; *Ziegel* [27].

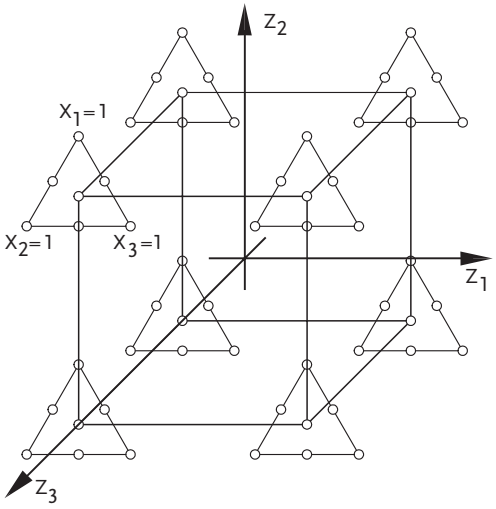

Figure 3.33a Simplex-centroid design in each point of a 2^3 full factorial experiment

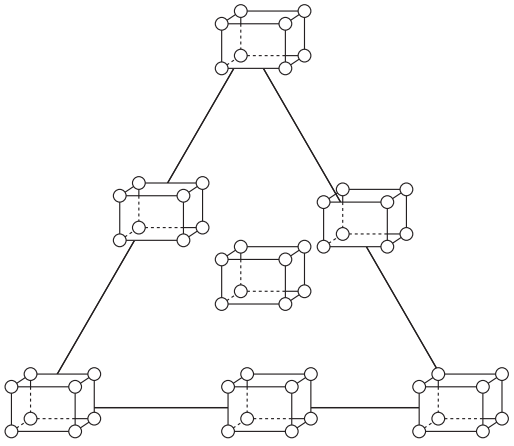

Figure 3.33b 2^3 Factorial design in each point of a simplex-centroid design

The design of experiment with its outcomes according to *Cornell's* [23] researches is given in Table 3.54. The patty composition was defined by proportions of X_1, X_2, X_3. Seven compositions are given in the above table, composition (1/2, 1/2, 0) means a mixture of 50% X_1, 50% X_2 and 0% X_3. Eight combinations of process factors Z_1, Z_2 and Z_3 are defined in coded values (-1,+1). Coded values of process factors are determined in this way:

$$Z_1 = \frac{z_1 - 204}{14}; \qquad Z_2 = \frac{z_2 - 32.5}{7.5}; \qquad Z_3 = \frac{z_3 - 32.5}{7.5} \tag{3.134}$$

Table 3.54 Simplex-centroid × full factorial design

Coded values of process factors			Proportion of components (x_1, x_2, x_3)						
Z_1	Z_2	Z_3	(1,0,0)	(0,1,0)	(0,0,1)	(1/2,1/2,0)	(1/2,0,1/2)	(0,1/2,1/2)	(1/3,1/3,1/3)
−	−	−	1.84	0.67	1.51	1.29	1.42	1.16	1.59
+	−	−	2.86	1.10	1.60	1.53	1.81	1.50	1.68
−	+	−	3.01	1.21	2.32	1.93	2.57	1.83	1.94
+	+	−	4.13	1.67	2.57	2.26	3.15	2.22	2.60
−	−	+	1.65	0.58	1.21	1.18	1.45	1.07	1.41
+	−	+	2.32	0.97	2.12	1.45	1.93	1.28	1.54
−	+	+	3.04	1.16	2.00	1.85	2.39	1.60	2.05
+	+	+	4.13	1.30	2.75	2.06	2.82	2.10	2.32

The system response is texture measured as the pressure in milligrams required to puncture the patty in a standardized setup. The response was obtained by replicating trials. The complete 56 coefficients regression model is obtained by multiplying each member in a seven-coefficient model for the mixture composition.

$$y = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3 \quad (3.135)$$

with each of eight coefficients in the regression model of process factors.

$$y = \alpha_0 + \alpha_1 Z_1 + \alpha_2 Z_2 + \alpha_3 Z_3 + \alpha_{12} Z_1 Z_2 + \alpha_{13} Z_1 Z_3 + \alpha_{23} Z_2 Z_3 + \alpha_{123} Z_1 Z_2 Z_3 + \varepsilon \quad (3.136)$$

The result of multiplication is the regression model:

$$\begin{aligned}\widehat{y}(X,Z) = &\sum_{i=1}^{3}\left[b_i^0 + \sum_{l=1}^{3} b_i^{\ell} Z_l + \sum_{l\prec m\le 3}^{3} b_i^{\ell m} Z_l Z_m + b_i^{123} Z_1 Z_2 Z_3\right] X_i \\ &+ \sum_{i\prec j\le 3}\left[b_{ij}^0 + \sum_{l=1}^{3} b_{ij}^{\ell} Z_l + \sum_{l\prec m\le 3}^{3} b_{ij}^{\ell m} Z_l Z_m + b_{ij}^{123} Z_1 Z_2 Z_3\right] X_i X_j \\ &+ \left[b_{123}^0 + \sum_{l=1}^{3} b_{123}^{\ell} Z_l + \sum_{l\prec m\le 3}^{3} b_{123}^{\ell m} Z_l Z_m + b_{123}^{123} Z_1 Z_2 Z_3\right] X_1 X_2 X_3\end{aligned} \quad (3.137)$$

Estimates of regression coefficients based on results from Table 3.54 are given in Table 3.55. It should be mentioned that coefficient notation b_{ij}^{ℓ} corresponds to *Scheffe* notation, and index ℓ is conected to process factors, while index ij has to do with mixture of components.

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Table 3.55 Estimates b_{sr}^{ℓ} and associated standard errors

Marks	Averages	Z_1	Z_2	Z_3	Z_1Z_2	Z_1Z_3	Z_2Z_3	$Z_1Z_2Z_3$	SE
X_1	2.87*	0.49*	0.71*	-0.09	0.07	-0.05	0.10	0.04	0.05
X_2	1.08*	0.18*	0.25*	-0.08	-0.03	-0.05	-0.03	-0.04	0.05
X_3	2.01*	0.25*	0.40*	0.01	0.00	0.17*	-0.05	-0.04	0.05
X_1X_2	-1.14*	-0.81*	-0.59	0.10	-0.06	0.14	-0.19	-0.09	0.23
X_1X_3	-1.00*	-0.54	-0.01	-0.03	-0.06	-0.27	-0.43	-0.12	0.23
X_2X_3	0.20	-0.14	0.07	-0.19	0.23	-0.25	0.12	0.27	0.23
$X_1X_2X_3$	3.18	0.07	-1.41	0.11	1.74	-0.71	1.77	-1.33	1.65

* Statistically significant regression coefficients with 99% confidence

Table 3.56 Estimates b_{sr}^{ℓ} for a reduced regression model

Marks	Averages	Z_1	Z_2	Z_3	Z_1Z_2	Z_1Z_3	Z_2Z_3	$Z_1Z_2Z_3$	SE
X_1	2.87	0.49	0.70	-0.06*	–	-0.06*	–	–	0.04
X_2	1.10	0.17	0.26	-0.06*	–	-0.06*	–	–	0.04
X_3	2.03	0.24	0.39	-0.06*	–	0.12*	–	–	0.04
X_1X_2	-1.17	-0.80	-0.66	–	–	–	–	–	0.20
X_1X_3	-1.03	-0.52	–	–	–	–	–	–	0.20
X_2X_3	–	–	–	–	–	–	–	–	–
$X_1X_2X_3$	3.67	–	–	–	–	–	–	–	1.32

* $b_1^3=b_2^3=b_3^3=0.06$; SE=0.02 $b_1^{13}=b_2^{13}=-0.06$; SE=0.02 $b_3^{13}=0.12$; SE=0.04

Most of the regression coefficients in Table 3.55 are not statistically significant so that the t-test suggest that a reduced regression model may adequately describe experimental outcomes. Regresssion coefficients and regression analysis for a reduced regression model are given in the work of *Gorman* and *Cornell* [24] and its summarized results in Table 3.56.

All regression coefficients in Table 3.56 are statistically significant with 99% confidence.

In the case of constraints on proportions of components the approach is known, simplex-centroid designs are constructed with coded or pseudocomponents [23]. Coded factors in this case are linear functions of real component proportions, and data analysis is not much more complicated in that case. If upper and lower constraints (bounds) are placed on some of the X_i resulting in a factor space whose shape is different from the simplex, then the formulas for estimating the model coefficients are not easily expressible. In the simplex-centroid $\times\ 2^3$ full factorial design or simplex-lattice $\times\ 2^n$ design [5], the number of points increases rapidly with increasing numbers of mixture components and/or process factors. In such situations, instead of full factorial we use fractional factorial experiments. The number of experimental trials required for studying the combined effects of the mixture com-

ponents and process factors depends on the form of the *combined* model to be fitted in the X_i's and Z_i's. As an alternative to fitting the complete model containing 2^{q+n}-2^n terms, let us define the *reduced combined* model with fewer than 2^{q+n}-2^n terms. The reduced model can be written using the following abbreviated models:

$$\left.\begin{aligned}
M_{1+1} &: \sum_{i=1}^{q} \beta_i^0 X_i + \sum_{\ell=1}^{n} \sum_{i=1}^{q} \beta_i^{\ell} X_i Z_\ell \\
M_{1+2} &: \sum_{\ell \prec m \le n} \sum_{i=1}^{q} \beta_i^{\ell m} X_i Z_\ell Z_m \\
M_{1+3} &: \sum_{\ell \prec m \prec p \le n} \sum_{i=1}^{q} \beta_i^{\ell m p} X_i Z_\ell Z_m Z_p \\
&\vdots \\
M_{1+n} &: \sum_{i=1}^{q} \beta_i^{12\ldots n} X_i Z_1 Z_2 \ldots Z_n
\end{aligned}\right\} \tag{3.138}$$

$$\left.\begin{aligned}
M_{2+1} &: \sum_{i \prec j \le q} \beta_{ij}^0 X_i X_j + \sum_{\ell=1}^{n} \sum_{i \prec j \le q} \beta_{ij}^{\ell} X_i X_j Z_\ell \\
M_{2+2} &: \sum_{\ell \prec m \le n} \sum_{i \prec j \le q} \beta_{ij}^{\ell m} X_i X_j Z_\ell Z_m \\
&\vdots \\
M_{3+1} &: \sum_{i \prec j \prec k \le q} \beta_{ijk}^0 X_i X_j X_k + \sum_{\ell=1}^{n} \sum_{i \prec j \prec k \le q} \beta_{ijk}^{\ell} X_i X_j X_k Z_\ell \\
&\vdots \\
M_{3+n} &: \sum_{i \prec j \prec k \le q} \beta_{ijk}^{12\ldots n} X_i X_j X_k Z_1 Z_2 \ldots Z_n
\end{aligned}\right\} \tag{3.139}$$

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where:
the subscript r+s in M_{r+s} refers to the inclusion of terms of degrees r and s in the X_i's and Z_i's, respectively.

The model M_{r+1} contains the r-th degree term in the mixture components only along with the product of this term with the first degree terms in the Z_i's. For example, a planar or first-degree model in the mixture components, and a main effects only model in the process variables, is $y=M_{1+1}+\varepsilon$. A planar model in the X_i's, combined with a main effect plus first-order interaction effects model in the Z_i's, would be $y=M_{1+1}+M_{1+2}+\varepsilon$. The model containing up to quadratic blending terms by main effects in the Z_i's is defined as $y=M_{1+1}+M_{2+1}+\varepsilon$. This continues, up to the complete 2^{q+n}-2^n term model that is defined as:

$$y = M_{1+1} + M_{2+1} + \ldots + M_{q+n} + \varepsilon$$

The choice of design arrangement, in terms of the number of points as well as the coordinate settings of the points, depends on the specific form of the model to be fitted. For the mixture components, if only the first-degree terms in the X_i are to be fitted, then just two values of X_i are required (say, X_i=0 and X_i=1). If the quadratic blending terms are to be fitted also, then the binary blends X_i=X_j=1/2, X_k=0, are required in addition to the settings X_i=0; 1. Similar arguments hold for the inclusion of the terms representing the different combinations of levels of the process variables. The number of combinations depends on the types of effects (main effects, two-factor interactions, and so on) that are to be estimated. In this section we shall be interested primarily in measuring the main effects of the process variables, particularly as they affect the blending properties of the mixture components. To begin investigation of the types of designs that are possible, we shall limit the discussion for the moment to the case of two mixture components q=2, whose proportions are denoted by X_1 and X_2, combined with three process factors n=3, which are represented by Z_1, Z_2, and Z_3. Also, for simplicity of illustration, let us write the model to be fitted as $y=M_{1+1}+M_{2+1}+\varepsilon$. Although simple in form, this model provides measures of the linear effects of the individual components, the quadratic (nonlinear) effect of blending the two components, and the main effects of the three process factors on the linear and nonlinear effects of the two components. To fit the model $y=M_{1+1}+M_{2+1}+\varepsilon$, data is collected for each of the three blends (X_1, X_2)=(1,0); (0,1); (1/2, 1/2) for each of the selected combinations of the levels of Z_1, Z_2 and Z_3. Let us denote the two mixture components whose proportions were represented previously by X_1, X_2 by A and B. The presence (absence) of the lower case letters a or b in the trial symbol represents the presence (absence) of the component. The three blends defined previously as (1,0); (0,1) and (1/2, 1/2) are now a, b and ab, respectively.

Let us denote the three process factors by D, E, F, where the presence (absence) of the lower case letters d, e or f in the trial designation indicates that the high (low) level of the factor is used. The eight combinations of factor levels are (1), d, e, de, f, df, ef and def. The trial designation ad means component A is present (but component B is not) at the high level of factor D and low levels of factors E and F. The trial designation abd means components A and B are present in a 1:1 blend at the high level of factor D and low levels of factors E and F.

Two possible designs that can be used for collecting data from which to estimate the coefficients for the model $y=M_{1+1}+M_{2+1}+\varepsilon$ are presented in Figs. 3.34 and 3.35.

In each design the number of combinations of the levels of the factors D, E and F is reduced to 4 from 2^3=8, by using a (2^{3-1}) half-fractional replica. In the design in Fig. 3.34, the fraction consisting of the process-factor level combinations d, e, f and def is set up at each point of composition. We denote this fraction of the full factorial design by I+DEF because the overall mean and the three-factor interaction effect DEF are measured jointly using the same linear combination of the observations. The main effect of factor D and EF interaction effect are estimated by the same contrast among the observations; the same can be said for effects E and DF, and F and DE. In the design in Fig. 3.35, the fraction I+DEF is set up at the pure blends [(X_1, X_2)=(1,0); (0,1)], but the opposite fraction, denoted by I-DEF, consisting of the level combinations (1), de, df and ef is set up at the (1/2, 1/2) blend of A and B.

	ad	abd	bd
	ae	abe	be
	af	abf	bf
	adef	abdef	bdef
	– o –	– o –	– o –
A:	1	1/2	0
B:	0	1/2	1
	I + DEF	I + DEF	I + DEF

Figure 3.34 The same 2^{3-1} fractional replicate design (i+def) in the process factors d, e, and f set up at the three points of composition (a,ab,b)

	ad	ab	bd
	ae	abde	be
	af	abdf	bf
	adef	abef	bdef
	– o –	– o –	– o –
A:	1	1/2	0
B:	0	1/2	1
	I + DEF	I – DEF	I + DEF

Figure 3.35 Different 2^{3-1} fractional replicate designs (i±def) in the process factors d, e and f set up at the three points of composition (a,ab,b)

In estimating the coefficients $\beta_1^{\ell}, \beta_2^{\ell}, \beta_{12}^{\ell}$ in the model $y=M_{1+1}+M_{2+1}+\varepsilon$, two questions of interest arise:

1) Are the calculating formulas for the estimates of the model coefficients the same with both designs?
2) How, if at all, do the variance and bias of the coefficient estimates differ for the two designs?

With the design in Figs. 3.34 and 3.35, the calculating formulas for the coefficient estimates $b_1\ell$ and $b_2\ell$ (ℓ=0, 1, 2 and 3) involve simple linear combinations of the average responses at the design points. Denoting the observed response value to the trial ad by ad for example, the estimates of the linear and main effect coefficients are calculated as:

$$b_1^0 = \frac{1}{4}(ad + ae + af + adef) = \bar{y} \text{ for } X_1 = 1 \tag{3.140}$$

$$b_1^1 = \frac{1}{2}\left[\frac{ad+adef}{2} - \frac{ae+af}{2}\right] = D^+ \text{ for } X_1 = 1 \tag{3.141}$$

$$b_1^2 = \frac{1}{2}\left[\frac{ae+adef}{2} - \frac{ad+af}{2}\right] = E^+ \text{ for } X_1 = 1 \tag{3.142}$$

$$b_1^3 = \frac{1}{2}\left[\frac{af+adef}{2} - \frac{ad+ae}{2}\right] = F^+ \text{ for } X_1 = 1 \tag{3.143}$$

$$b_2^0 = \frac{1}{4}(bd + be + bf + bdef) = \bar{y} \text{ for } X_2 = 1 \tag{3.144}$$

$$b_2^1 = D^+ \quad \text{for } X_2 = 1; \quad b_2^2 = E^+ \quad \text{for } X_2 = 1; \quad b_2^3 = F^+ \quad \text{for } X_2 = 1 \tag{3.145}$$

$$b_{12}^0 = 4[\bar{y} \text{ for } X_1 = X_2 = 1/2] - 2\{[\bar{y} \text{ for } X_1 = 1] + [\bar{y} \text{ for } X_2 = 1]\} \tag{3.146}$$

$$b_{12}^1 = 4[\text{main effect } D \text{ for } X_1 = X_2 = 1/2] - 2\{[\text{main effect } D \text{ for } X_1 = 1] + [\text{main effect } D \text{ for } X_2 = 1]\} \tag{3.147}$$

For the design in Fig. 3.34:

$$b_{12}^0 = 4\left[\frac{1}{4}(abd + abe + abf + abdef)\right] - 2\left[\frac{1}{4}(ad + ae + af + adef) + \frac{1}{4}(bd + be + bf + bdef)\right] \tag{3.148}$$

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$$
\begin{aligned}
b_{12}^{1} &= 4\left[\frac{1}{2}\left(\frac{abd+abdef}{2}-\frac{abe+abf}{2}\right)\right] \\
&- 2\left[\frac{1}{2}\left(\frac{ad+adef}{2}-\frac{ae+af}{2}\right)+\frac{1}{2}\left(\frac{bd+bdef}{2}-\frac{be+bf}{2}\right)\right] \\
&= 4\left[D^{+} \text{ for } X_1 = X_2 = 1/2\right] - 2\left\{\left[D^{+} \text{ for } X_1 = 1\right] + \left[D^{+} \text{ for } X_2 = 1\right]\right\}
\end{aligned}
\tag{3.149}
$$

For the design in Fig. 3.35:

$$
\begin{aligned}
b_{12}^{0} &= 4\left[\frac{1}{4}(ab+abde+abdf+abef)\right] \\
&-2\left[\frac{1}{4}(ad+ae+af+adef)+\frac{1}{4}(bd+be+bf+bdef)\right]
\end{aligned}
\tag{3.150}
$$

$$
\begin{aligned}
b_{12}^{1} &= 4\left[\frac{1}{2}\left(\frac{abde+abdf}{2}-\frac{ab+abef}{2}\right)\right] \\
&- 2\left[\frac{1}{2}\left(\frac{ad+adef}{2}-\frac{ae+af}{2}\right)+\frac{1}{2}\left(\frac{bd+bdef}{2}-\frac{be+bf}{2}\right)\right] \\
&= 4[D^{-} \text{ for } X_1 = X_2 = 1/2] - 2\left\{\left[D^{+} \text{ for } X_1 = 1\right] + \left[D^{+} \text{ for } X_2 = 1\right]\right\}
\end{aligned}
\tag{3.151}
$$

Let us illustrate numerically the calculation formulas for the estimates $b_1\ell$,$b_2\ell$,$b_{12}\ell$, with the designs in Figs. 3.34 and 3.35 using the data in Table 3.54: The first term in $b_1{}^0$ is ad, which corresponds to the value 2.86 found in the (1, -1, -1) row of column (1, 0, 0). This is the observed response when D is at the high level, and E and F are at their low levels, which corresponds to (Z_1, Z_2 , Z_3) = (1, -1, -1). The a indicates that only component A is present, so one uses the (1, 0, 0) column. Similarly, one obtains for either design

$$
\begin{aligned}
b_1^0 &= \frac{1}{4}(2.86+3.01+1.65+4.13) = 2.91; \\
b_1^1 &= \frac{1}{2}\left(\frac{2.86+4.13}{2}-\frac{3.01+1.65}{2}\right) = 0.58 \\
b_1^2 &= 0.66; \quad b_1^3 = -0.02; \\
b_2^0 &= \frac{1}{4}(1.10+1.21+0.58+1.30) = 1.05; \\
b_2^1 &= \frac{1}{2}\left(\frac{1.10+1.30}{2}-\frac{1.21+0.58}{2}\right) = 0.15 \\
b_2^2 &= 0.21; \quad b_2^3 = -0.11;
\end{aligned}
$$

For design, Fig. 3.34:

$$
\begin{aligned}
b_{12}^{0} &= 4\left[\frac{1}{4}(1.53+1.93+1.18+2.06)\right] \\
&\quad - 2\left[\frac{1}{4}(2.86+3.01+1.65+4.13)+\frac{1}{4}(1.10+1.21+0.58+1.30)\right] \\
&\quad = 6.70 - 2(2.91+1.05) = -1.22 \\
b_{12}^{1} &= 4\left[\frac{1}{2}\left(\frac{1.53+2.06}{2}-\frac{1.93+1.18}{2}\right)\right] \\
&\quad - 2\left[\frac{1}{2}\left(\frac{2.86+4.13}{2}-\frac{3.01+1.65}{2}\right)+\frac{1}{2}\left(\frac{1.10+1.30}{2}-\frac{1.21+0.58}{2}\right)\right] \\
&\quad = 0.48 - 2(0.58+0.15) = -0.98 \\
b_{12}^{2} &= -0.46; b_{12}^{3} = 0.04.
\end{aligned}
$$

For design, Fig. 3.35:

$$b_{12}^{0} = 4\left[\frac{1}{4}(1.29 + 2.26 + 1.45 + 1.85)\right] - 2\left[\frac{1}{4}(2.86 + 3.01 + 1.65 + 4.13) + \frac{1}{4}(1.10 + 1.21 + 0.58 + 1.30)\right] = -1.07$$

$$b_{12}^{1} = 4\left[\frac{1}{2}\left(\frac{2.26 + 1.45}{2} - \frac{1.29 + 1.85}{2}\right)\right] - 2\left[\frac{1}{2}\left(\frac{2.86 + 4.13}{2} - \frac{3.01 + 1.65}{2}\right) + \frac{1}{2}\left(\frac{1.10 + 1.30}{2} - \frac{1.21 + 0.58}{2}\right)\right] = -0.89$$

$b_{12}^{2} = -0.37; b_{12}^{3} = 0.01.$

The regression model for design Fig. 3.34 is:

$$\begin{aligned}\widehat{y}(X, Z) = {} & (2.91 + 0.58Z_1 + 0.66Z_2 - 0.02Z_3)X_1 \\ & + (1.05 + 0.15Z_1 + 0.21Z_2 - 0.11Z_3)X_2 \\ & + (-1.22 - 0.98Z_1 - 0.46Z_2 + 0.04Z_3)X_1X_2 \end{aligned} \tag{3.152}$$

The regression model for design Fig. 3.35 is:

$$\begin{aligned}\widehat{y}(X, Z) = {} & (2.91 + 0.58Z_1 + 0.66Z_2 - 0.02Z_3)X_1 \\ & + (1.05 + 0.15Z_1 + 0.21Z_2 - 0.11Z_3)X_2 \\ & + (-1.07 - 0.89Z_1 - 0.37Z_2 + 0.01Z_3)X_1X_2 \end{aligned} \tag{3.153}$$

The closeness of the values of the estimates $b_1^{\ell}, b_2^{\ell}, b_{12}^{\ell}$, obtained with the designs in Figs. 3.34 and 3.35, to the values of the corresponding estimates in Table 3.55 obtained from the full factorial design is evidence of the absence of interaction effects among the process factors (particularly with respect to the effect of the two components whose proportions are denoted by X_1 and X_2). The approximate equality of the values of the estimates obtained with the fractional and full factorial designs lends support to our decision to consider the use of fractional designs in the process factors when interactions among these factors are negligible.

Thus far we have discussed only the two components *simplex-centroid×three process factor full factorial design*. By keeping the design and model simple, the calculating formulas for the coefficient estimates were easily written for the two fractional replica designs presented. When more than two mixture components are combined with more than three process factors and the simplex-centroid design is joined with a full factorial design or some fraction of the full factorial, similar formulas involving the trials combinations can be set up for estimating the model coefficients. For example, Table 3.57 lists several possible fractional designs (denoted by roman numerals I, II, III, IV and V) along with the corresponding estimable coefficients in the fitted models for experiments where three components are combined with three process factors. A strategy that can be used to generate fractions of the simplex-centroid $\times 2^n$ designs, and, that was in fact used to generate the fractional designs in Table 3.57, is given in *Scheffe's* work [6].

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When three components X_1, X_2 and X_3 are combined with three process factors Z_1, Z_2 and Z_3, two possible designs, similar to designs in Figs. 3.34 and 3.35, are presented in Figs. 3.36a and 3.36b.

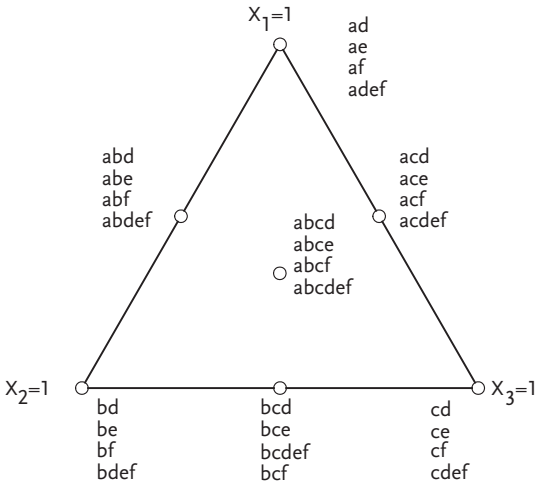


Figure 3.36a Fractional design in each point of a simplex-centroid design

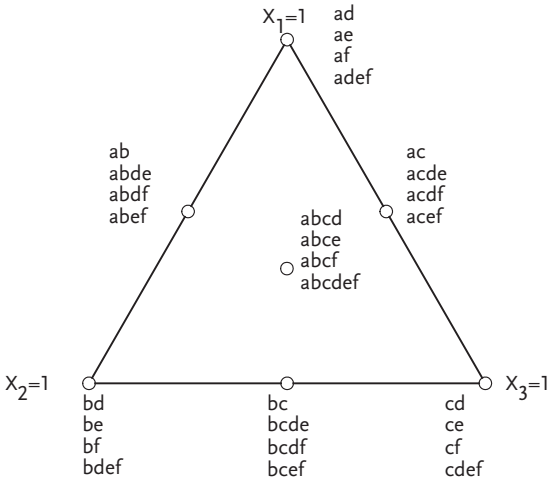
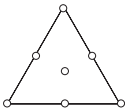
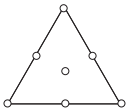
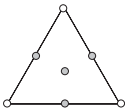
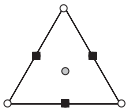
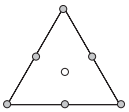
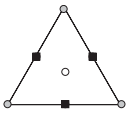
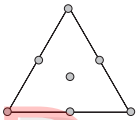
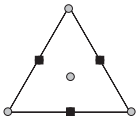
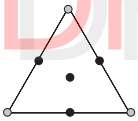
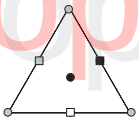
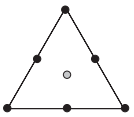
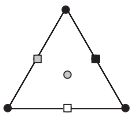


Figure 3.36b Fractional design in each point of a simplex-centroid design

Tabel 3.57 Simplex-centroid × full factorial design (q=3, n=3)

Design	N	Design a	Design b	Regression coefficients
0	56			$\widehat{y} = M_{1+1} + M_{2+1} + \cdots + M_{q+n}$
I	40			$\left[\beta_i^{\ell}, \beta_{ij}^{\ell}, \beta_{123}^{\ell}, 1 \le i \prec j \prec 3, \ell = 0,1,2,3\right]$ $\left\{ \begin{matrix} \left(\beta_i^{12}, \beta_i^{13}, \beta_i^{23}, \beta_i^{123}, 1 \le i \le 3\right) \text{or} \\ (\alpha_{12}, \alpha_{13}, \alpha_{23}, \alpha_{123}) \end{matrix} \right\}$
II	32			$\left[\beta_i^0, \beta_i^1, \beta_i^2, \beta_i^3, i = 1,2,3\right]$, $\left[\beta_{ij}^{\ell}, 1 \le i \prec j \le 3, \ell = 0,1,2,3\right]$, $\left[\beta_{123}^{\ell}, \ell = 0,1,2,3\right]$ $\left\{ \begin{matrix} \left[\beta_{123}^{\ell m}, 1 \le \ell \prec m \le 3, \beta_{123}^{123}\right] \text{or} \\ [\alpha_{12}, \alpha_{13}, \alpha_{23}, \alpha_{123}] \end{matrix} \right\}$
III	28			$\left[\beta_i^0, \beta_i^1, \beta_i^2, \beta_i^3, i = 1,2,3\right]$, $\left[\beta_{ij}^{\ell}, 1 \le i \prec j \le 3, \ell = 0,1,2,3\right]$, $\left[\beta_{123}^{\ell}, \ell = 0,1,2,3\right]$
IV	20			$\left[\beta_i^0, \beta_i^1, \beta_i^2, \beta_i^3, i = 1,2,3\right]$, $\left[\beta_{12}^0, \beta_{13}^0, \beta_{23}^0, \beta_{123}^0\right]$ + 4 degrees of freedom
V	16			$\left[\beta_1^0, \beta_2^0, \beta_3^0, \beta_{12}^0, \beta_{13}^0, \beta_{23}^0, \beta_{123}^0\right]$, [process factors main effects] + 6 degrees of freedom

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◯ - PFE 2^3 ● - (1), def ◯ - (1),de,df,ef □ - e,df ▭ - d,ef ■ - d,e,f,def

The given designs are used for fitting a three-component simplex-centroid (or an incomplete cube model) with main effects of process factors:

$$\widehat{y} = \sum_{i=1}^{3} \beta_i^0 X_i + \sum_{i \prec j \le 3} \beta_{ij}^0 X_i X_j + \beta_{123}^0 X_1 X_2 X_3 + \sum_{\ell=1}^{3} \left[\sum_{i=1}^{3} \beta_i^{\ell} X_i + \sum_{i \prec j \le 3} \beta_{ij}^{\ell} X_i X_j + \beta_{123}^{\ell} X_1 X_2 X_3 \right] Z_l + \varepsilon \qquad (3.154)$$

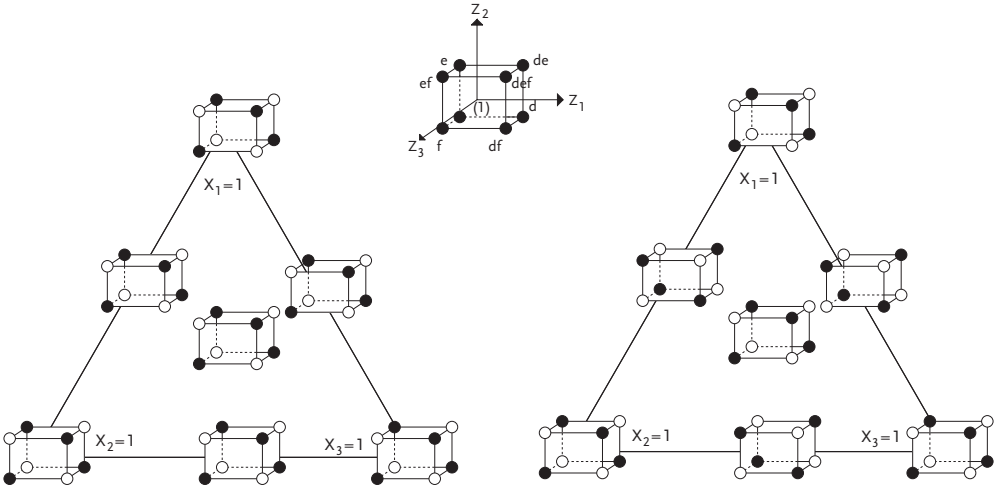

Figure 3.37 Fractional simplex designs by Figs. 3.36a and 3.36b

3.10 Examples of Complex Optimizations of Mixture Problems

Example I [28]

A nine-component composition has been tested in developing a new composite material. Three components were the binder of the composite material: polyester EPX-279-1, polyester EPX-187-3 and styrene. These materials have been used as fillers: ash, marble powder, glass microspheres, saran microspheres, wollastonite and powder made by grinding shells. Component properties with variation range of their proportions are given in Table 3.58.

Table 3.58 Component properties

Component	Denotation	Density g/cm³	Av. Diameter μm	Price ¢/lb	Price ¢/l	Variation Range
Ash	X_1	2.580	4.5	1.97	11.20	1–5
Marble	X_2	2.710	40.0	0.50	3.00	1–25
Glass	X_3	0.340	62.0	69.00	52.00	1–25
Saran	X_4	0.032	30.0	350.00	26.30	1–25
Wollastonite	X_5	2.890	30.0	1.85	11.80	1–25
Shell powder	X_6	1.300	125.0	2.50	7.16	5–29
Polyester epx-279-1	X_7	1.148	–	26.80	67.80	58–80
Polyester epx-187-3	X_8	1.335	–	18.00	56.50	5–29
Styren	X_9	0.900	–	11.50	22.81	5–29

To obtain a second-order regression model, an experiment by extreme vertices design has been done. The experiment included 45 trials including a simplex center for a check of lack of fit of the regression model and an estimate of experimental error, as shown in Table 3.59.

Each trial has been done by mixing components in a corresponding ratio on a vertical mixer. The produced mass is poured into corresponding molds, from which it is taken out after 800 s. Samples are then cured in a drying room for 20 h at 70 °C. By testing samples, their density, tensile strength, bending strength, pressing strength, elasticity module, bending modulus and elongation at break are determined. Besides, based on composition, the expenditure for each trial has been calculated. The outcomes of the experiment are in Table 3.60. Fitting experimental outcomes has been done by a second-order polynomial:

$$y = B_0 + \sum_{i=1}^{8} B_i X_i + \sum_{i=1}^{8} B_{ii} X_i^2 + \sum_{i=1}^{7} \sum_{j=1+1}^{8} B_{ij} X_i X_j \tag{3.155}$$

It should be noted that regression coefficients have been calculated for all property measurements except density. Calculations have been done on a computer due to a large number of components and properties, Table 3.61. The density of the composite material has been fitted with a linear regression:

$$\rho = B_0 + \sum_{i=1}^{8} B_i X_i \tag{3.156}$$

Due to the specific application of the composite material, limitations on measured properties have been imposed in one case:

$\rho \leq 1.2\,\mathrm{g/cm^3}$; $\sigma_{z\sim} \geq 3000\,\mathrm{psi}$; $\sigma_s \geq 2500\,\mathrm{psi}$

$\sigma_p \geq 5000\,\mathrm{psi}$; $E \geq 4 \times 10^5\,\mathrm{psi}$; $E_s \geq 4 \times 10^5\,\mathrm{psi}$; $\varepsilon \geq 15\%$

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Apart from the mentioned limitations on properties, limitations on components have also been imposed:

$$\sum_{i=1}^{6} X_i \leq 0.55; \qquad 1 - \sum_{i=1}^{8} X_i \leq \frac{1}{3}(X_7 + X_8)$$

By application of computer and associated programs for optimization with limitations-*compromising optimization*, the composition of the composite has been determined:

X_1=0.050; X_2=0.025; X_3=0.000; X_4=0.200; X_5=0.013; X_6=0.235;

$X_7 = 0.115$; $X_8 = 0.245$; $X_9 = 0.117$; $\sum_{i=1}^{6} X_i = 0.523$; $X_7 + X_8 = 0.360$.

Table 3.59 Extreme vertices design

N	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9
1	0.25	0.01	0.01	0.01	0.01	0.05	0.56	0.05	0.05
2	0.13	0.13	0.01	0.01	0.01	0.05	0.56	0.05	0.05
3	0.13	0.01	0.13	0.01	0.01	0.05	0.56	0.05	0.05
4	0.13	0.01	0.01	0.13	0.01	0.05	0.56	0.05	0.05
5	0.13	0.01	0.01	0.01	0.13	0.05	0.56	0.05	0.05
6	0.13	0.01	0.01	0.01	0.01	0.17	0.56	0.05	0.05
7	0.13	0.01	0.01	0.01	0.01	0.05	0.68	0.05	0.05
8	0.13	0.01	0.01	0.01	0.01	0.05	0.56	0.17	0.05
9	0.13	0.01	0.01	0.01	0.01	0.05	0.56	0.05	0.17
10	0.01	0.25	0.01	0.01	0.01	0.05	0.56	0.05	0.05
11	0.01	0.13	0.13	0.01	0.01	0.05	0.56	0.05	0.05
12	0.01	0.13	0.01	0.01	0.01	0.05	0.56	0.05	0.05
13	0.01	0.13	0.01	0.01	0.13	0.05	0.56	0.05	0.05
14	0.01	0.13	0.01	0.13	0.01	0.17	0.56	0.05	0.05
15	0.01	0.13	0.01	0.01	0.01	0.05	0.68	0.05	0.05
16	0.01	0.13	0.01	0.01	0.01	0.05	0.56	0.17	0.05
17	0.01	0.13	0.01	0.01	0.01	0.05	0.56	0.05	0.17
18	0.01	0.01	0.25	0.01	0.01	0.05	0.56	0.05	0.05
19	0.01	0.01	0.13	0.13	0.01	0.05	0.56	0.05	0.05
20	0.01	0.01	0.13	0.01	0.13	0.05	0.56	0.05	0.05
21	0.01	0.01	0.13	0.01	0.01	0.17	0.56	0.05	0.05
22	0.01	0.01	0.13	0.01	0.01	0.05	0.68	0.05	0.05
23	0.01	0.01	0.13	0.01	0.01	0.05	0.56	0.17	0.05
24	0.01	0.01	0.13	0.01	0.01	0.05	0.56	0.05	0.17
25	0.01	0.01	0.01	0.25	0.01	0.05	0.56	0.05	0.05
26	0.01	0.01	0.01	0.13	0.13	0.05	0.56	0.05	0.05
27	0.01	0.01	0.01	0.13	0.01	0.17	0.56	0.05	0.05
28	0.01	0.01	0.01	0.13	0.01	0.05	0.68	0.05	0.05
29	0.01	0.01	0.01	0.13	0.01	0.05	0.56	0.17	0.05
30	0.01	0.01	0.01	0.13	0.01	0.05	0.56	0.05	0.17
31	0.01	0.01	0.01	0.01	0.25	0.05	0.56	0.05	0.05
32	0.01	0.01	0.01	0.01	0.13	0.17	0.56	0.05	0.05
33	0.01	0.01	0.01	0.01	0.13	0.05	0.68	0.05	0.05
34	0.01	0.01	0.01	0.01	0.13	0.05	0.56	0.17	0.05
35	0.01	0.01	0.01	0.01	0.13	0.05	0.56	0.05	0.17
36	0.01	0.01	0.01	0.01	0.01	0.29	0.56	0.05	0.05
37	0.01	0.01	0.01	0.01	0.01	0.17	0.68	0.05	0.05
38	0.01	0.01	0.01	0.01	0.01	0.17	0.56	0.17	0.05
39	0.01	0.01	0.01	0.01	0.01	0.17	0.56	0.05	0.17
40	0.01	0.01	0.01	0.01	0.01	0.05	0.80	0.05	0.05

Table 3.59 (continued)

N	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9
41	0.01	0.01	0.01	0.01	0.01	0.05	0.68	0.17	0.05
42	0.01	0.01	0.01	0.01	0.01	0.05	0.68	0.05	0.17
43	0.01	0.01	0.01	0.01	0.01	0.05	0.56	0.29	0.05
44	0.01	0.01	0.01	0.01	0.01	0.05	0.56	0.17	0.17
45	0.01	0.01	0.01	0.01	0.01	0.05	0.56	0.05	0.29
46	0.0367	0.0367	0.0367	0.0367	0.0367	0.0767	0.5867	0.0767	0.0767
47	0.0367	0.0367	0.0367	0.0367	0.0367	0.0767	0.5867	0.0767	0.0767
48	0.0367	0.0367	0.0367	0.0367	0.0367	0.0767	0.5867	0.0767	0.0767

Table 3.60 Measured properties

N	ρ g/cm^3	σ_{z-} 10^{-3} psi	σ_s 10^{-3} psi	σ_p 10^{-3} psi	E 10^{-3} psi	E_s 10^{-3} psi	ε %	Price ¢/l
1	1.524	1.790	5.066	6.992	96.772	75.821	6.25	46.03
2	1.540	2.174	5.746	6.995	144.436	98.033	3.50	45.05
3	1.255	1.992	5.158	6.744	150.662	200.548	3.75	50.93
4	1.218	1.810	4.812	7.759	104.285	73.772	4.75	47.84
5	1.561	2.547	6.320	6.841	192.286	238.093	2.75	46.10
6	1.370	2.619	6.527	8.425	176.678	255.389	2.83	45.56
7	1.353	1.742	5.350	9.087	76.219	119.019	13.00	52.82
8	1.375	2.740	6.457	10.320	170.477	203.992	3.16	51.47
9	1.323	2.647	7.008	11.265	158.520	196.839	4.50	47.44
10	1.556	1.756	4.930	5.053	120.088	178.962	4.75	44.06
11	1.271	1.495	4.193	5.049	105.821	144.552	5.50	49.94
12	1.234	1.326	3.922	5.826	68.542	92.949	8.83	46.86
13	1.571	2.012	5.710	5.072	149.286	106.484	3.50	45.12
14	1.360	2.093	5.640	6.273	123.925	90.966	4.25	44.58
15	1.369	1.354	4.322	7.250	57.920	84.523	13.30	51.84
16	1.391	2.676	6.291	8.583	172.846	212.616	3.50	50.48
17	1.339	2.391	5.931	9.483	155.229	84.923	4.00	46.45
18	0.986	1.055	3.020	6.851	68.198	87.952	10.00	55.82
19	0.950	0.979	3.111	7.875	49.265	39.920	12.70	52.74
20	1.293	1.529	4.401	4.697	117.360	89.910	4.25	51.00
21	1.102	1.579	4.645	6.073	101.706	76.399	5.50	50.46
22	1.085	1.178	3.822	9.195	49.636	93.970	20.25	57.72
23	1.107	2.134	5.855	8.791	140.909	171.469	5.00	56.36
24	1.055	1.951	5.627	10.742	113.217	149.987	8.16	52.33
25	0.913	0.831	2.405	7.880	28.853	39.518	16.70	49.66
26	1.255	1.551	4.641	4.123	88.557	145.391	7.10	47.92
27	1.065	1.362	3.995	6.659	60.385	47.749	9.25	47.37

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Table 3.60 (continued)

N	ρ g/cm^3	$\sigma_{z\sim}$ 10^{-3} psi	σ_s 10^{-3} psi	σ_p 10^{-3} psi	E 10^{-3} psi	E_s 10^{-3} psi	ε %	Price ¢/l
28	1.047	1.170	3.550	9.325	36.676	22.903	20.00	54.64
29	1.070	2.074	5.458	9.936	98.425	117.795	6.10	53.28
30	1.018	2.096	5.961	14.849	101.368	121.118	11.70	49.25
31	1.599	1.701	6.842	5.523	113.891	92.042	4.75	46.18
32	1.408	2.061	5.786	6.140	151.090	233.562	2.75	45.63
33	1.390	1.488	4.923	7.655	69.508	127.688	11.75	52.90
34	1.413	2.648	6.893	8.795	188.910	238.930	2.75	51.54
35	1.361	2.683	6.832	8.820	177.011	228.251	3.83	47.51
36	1.218	2.230	4.952	7.444	125.650	167.862	4.75	45.09
37	1.200	1.742	5.723	8.605	65.118	118.473	15.67	52.35
38	1.221	2.967	7.502	11.180	168.391	209.106	3.25	51.00
39	1.170	2.837	7.476	12.713	147.005	187.590	6.58	46.96
40	1.181	1.376	4.225	13.302	35.650	47.539	28.10	59.62
41	1.204	2.465	6.813	16.235	106.444	121.308	9.83	58.26
42	1.152	2.156	7.116	10.277	93.620	127.054	24.30	54.23
43	1.266	3.671	8.489	20.086	183.699	233.242	3.00	56.90
44	1.174	3.725	8.810	17.021	184.266	188.491	5.17	52.87
45	1.122	3.467	8.203	17.576	163.933	205.205	7.50	48.84
46	1.259	2.222	5.554	8.330	138.991	81.023	4.50	50.25
47	1.259	2.101	5.449	9.419	130.262	78.496	5.50	50.25
48	1.259	2.089	5.560	9.391	133.316	78.339	5.25	50.25

Table 3.61 Regression coefficients

Regression coefficients	ρ	ε	σ_p	$\sigma_{z\sim}$	σ_s	E_s	E
B_0	1.13249	-114.19733	0.07269	0.01064	0.01384	0.56932	0.4745
B_1	1.37649	95.98950	-0.02732	0.00078	-0.00283	0.14554	1.79251
B_2	1.57042	167.01782	-0.05128	-0.00615	-0.01668	-5.75500	0.70308
B_3	-1.02582	-14.61224	-0.04799	-0.00967	-0.020115	-1.22441	-0.29696
B_4	-1.10084	109.44629	-0.03456	-0.00979	-0.02067	0.22113	-1.35077
B_5	1.58401	160.08569	-0.06056	-0.00244	-0.01579	1.72925	1.58983
B_6	0.27504	82.41577	-0.03130	-0.00383	0.00103	-0.63911	0.50197
B_7	0.08755	290.07031	-0.12926	-0.01465	-0.00056	-0.33234	-0.64604
B_8	0.34422	256.43359	-0.22232	-0.00569	-0.02062	-1.35132	0.43842
B_{11}	0.0	182.80933	-0.05150	-0.02704	-0.04074	-2.97189	-3.16558
B_{22}	0.0	39.53931	0.00961	-0.00091	0.01035	8.39853	-0.30142
B_{33}	0.0	84.78394	0.02581	0.00139	-0.00693	0.72176	-0.41278
B_{44}	0.0	137.30420	-0.00937	-0.00172	-0.01476	1.06233	0.57664

Table 3.61 (continued)

Regression coefficients	ρ	ε	σ_p	$\sigma_{z\sim}$	σ_s	E_s	E
B_{55}	0.0	119.84521	0.05115	-0.01412	0.03872	-4.55200	-2.13765
B_{66}	0.0	25.01221	-0.02219	-0.00174	-0.04392	0.89334	-0.21522
B_{77}	0.0	-113.98730	0.07174	0.00431	-0.01337	-0.23637	0.16125
B_{88}	0.0	37.80859	0.21372	0.00680	0.01492	3.09570	-0.41095
B_{12}	0.0	0.0	0.0	0.0	0.0	1.93091	0.0
B_{13}	0.0	0.0	0.0	0.0	0.0	4.53545	0.0
B_{14}	0.0	0.0	0.0	0.0	0.0	-2.24657	0.0
B_{15}	0.0	0.0	0.0	0.0	0.0	1.72719	0.0
B_{16}	0.0	0.0	0.0	0.0	0.0	5.74762	0.0
B_{17}	0.0	-270.89063	0.0	0.0	0.0	-0.56160	-2.24062
B_{18}	0.0	0.0	0.0	0.0	0.0	2.11084	0.0
B_{23}	0.0	0.0	0.0	0.0	0.0	8.43297	0.0
B_{24}	0.0	0.0	0.0	0.0	0.0	6.87164	0.0
B_{25}	0.0	0.0	0.0	0.0	0.0	0.37309	0.0
B_{26}	0.0	0.0	0.0	0.0	0.0	2.11751	0.0
B_{27}	0.0	-342.40527	0.0	0.0	0.0	4.83197	-1.45135
B_{28}	0.0	0.0	0.0	0.0	0.0	10.49878	0.0
B_{34}	0.0	0.0	0.0	0.0	0.0	-1.32475	0.0
B_{35}	0.0	0.0	0.0	0.0	0.0	-5.29202	0.0
B_{36}	0.0	0.0	0.0	0.0	0.0	-3.41042	0.0
B_{37}	0.0	0.0	0.0	0.0	0.0	0.98099	0.0
B_{38}	0.0	0.0	0.0	0.0	0.0	3.12524	0.0
B_{45}	0.0	0.0	0.0	0.0	0.0	0.58264	0.0
B_{46}	0.0	0.0	0.0	0.0	0.0	-3.37820	0.0
B_{47}	0.0	-196.06665	0.0	0.0	0.0	-1.92389	1.13045
B_{48}	0.0	0.0	0.0	0.0	0.0	1.41870	0.0
B_{56}	0.0	0.0	0.0	0.0	0.0	2.08801	0.0
B_{57}	0.0	-372.80078	0.0	0.0	0.0	2.08035	-2.25305
B_{58}	0.0	61.21167	0.00910	-0.01003	-0.00587	2.39307	0.276081
B_{67}	0.0	-188.13574	0.0	0.0	0.0	0.07410	-1.06024
B_{68}	0.0	0.0	0.0	0.0	0.0	3.12915	0.0
B_{78}	0.0	-520.73828	0.29166	2.00905	0.02934	0.20239	-0.40131

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Example II [14]

When changing technological parameters in the process of refining of raw benzene, composition of waste liquid is also changed. Waste liquid is often used for obtaining expensive materials characterized by their density and viscosity. Water content in such a liquid varies from 0 to 15%, and the contents of ashes does not exceed 10%. The research objective is to obtain a regression model that will adequately describe density and viscosity in the function of proportions of organic matter, water and ashes. Since proportions of components are limited, the experiment has been done by extreme vertices design. The local factor space is given in Table 3.62, and design matrix with outcomes of trials in Table 3.63.

Table 3.62 Local factor space

Components	Proportions of components		
	x_1	x_2	x_3
X_1	95	0	5
X_2	80	15	5
X_3	90	0	10

Table 3.63 Extreme vertices design

Response Mark	Design matrix			Density g/cm^3			Viscosity p		
	X_1	X_2	X_3	ρ_1	ρ_2	$\bar{\rho}$	ν_1	ν_2	$\bar{\nu}$
y_1	1	0	0	1.128	1.126	1.127	17.1	16.6	16.85
y_2	0	1	0	1.070	1.072	1.071	4.0	5.0	4.50
y_3	0	0	1	1.139	1.136	1.1375	21.0	19.1	20.05
y_{12}	0.5	0.5	0	1.112	1.114	1.113	12.6	12.8	12.70
y_{13}	0.5	0	0.5	1.135	1.130	1.1325	18.6	17.9	18.25
y_{23}	0	0.5	0.5	1.118	1.120	1.119	14.6	14.8	14.70
y_{123}*	1/3	1/3	1/3	1.122	1.123	1.1225	15.0	15.6	15.30

The geometrical interpretation of local factor space is given in Fig. 3.38.

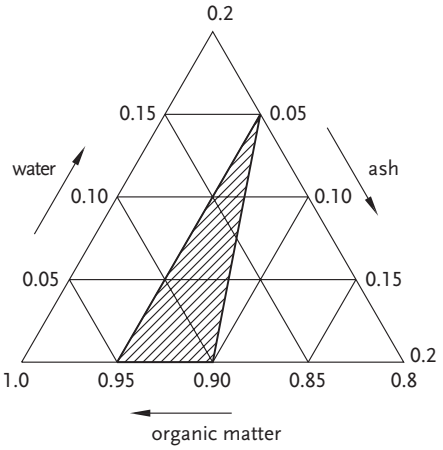

Figure 3.38 Local factor space

Connections between real and coded factors are given by these relations:

$x_1=0.95X_1 + 0.8X_2 + X_3$

$x_2=0.15X_2$

$x_3=0.05X_1 + 0.05X_2 + 0.1X_3$ (3.157)

By calculating regression coefficients, these second-order regression models have been obtained:

$$\widehat{\rho} = 1.127X_1 + 1.071X_2 + 1.138X_3 + 0.046X_1X_2 - 0.002X_1X_3 + 0.058X_2X_3 \quad (3.158)$$

$$\widehat{\nu} = 16.85X_1 + 4.5X_2 + 20.05X_3 + 8.1X_1X_2 + 9.7X_1X_3 - 0.8X_2X_3 \quad (3.159)$$

To check lack of fit of the obtained regression models, errors of the experiment have been calculated:

$$S_\rho^2 = 3.6 \times 10^{-6}; \quad S_\nu^2 = 0.422; \quad f = 7.$$

A check of lack of fit has been done in control point y_{123}. Calculated values of density and viscosity in control points have by Eqs. (3.158) and (3.159) these values, respectively are: ρ_{123}=1.123 and ν_{123}=15.69.

$$\Delta\rho_{123} = \left|\bar{\rho}_{123} - \widehat{\rho}_{123}\right| = |1.1225 - 1.1230| = 0.0005;$$

$$\Delta\nu_{123} = \left|\bar{\nu}_{123} - \widehat{\nu}_{123}\right| = |15.3 - 15.69| = 0.39$$

$$t_\rho = \frac{0.0005\sqrt{2}}{1.276\sqrt{3.6\times10^{-6}}} = 0.292; t_\nu = \frac{0.39\sqrt{2}}{1.276\sqrt{0.422}} = 0.655.$$

Since the tabular value is $t_{(0.025;7)}$=2.3646, calculated values t_R are smaller, so that regression models are adequate. The geometric interpretation of regression models as contour lines and in coded factors is given in Figs. 3.39 and 3.40.

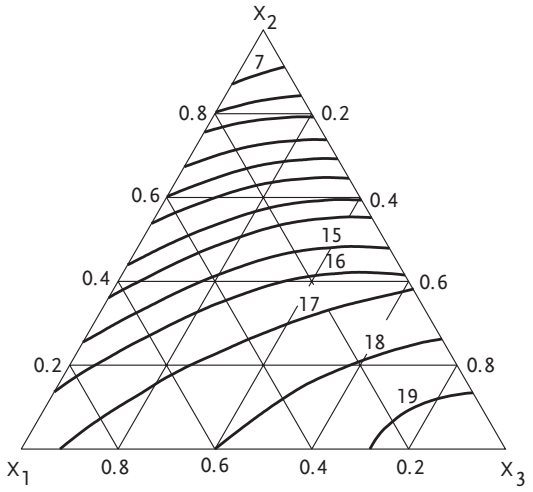

Figure 3.39 Viscosity contour lines

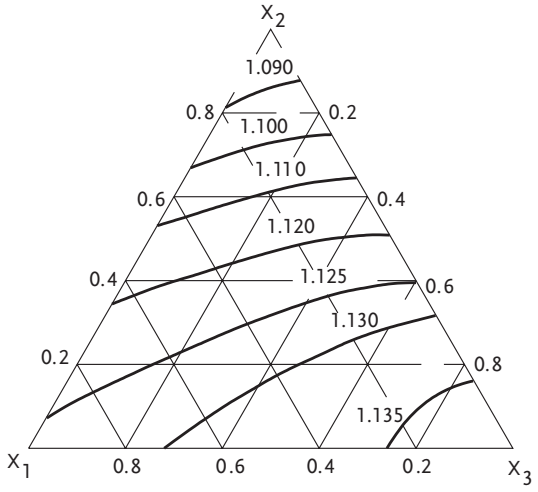

Figure 3.40 Density contour lines

Example III [29]

The research objective has been to define the durability of a coating depending on mixture composition Ni-Cr-B. Besides, one had to determine the optimal composition of the given three-component mixture. Since there is a linear correlation between resistance on wear-out and hardness of coating, Rockwell hardness *(HRC)* has been chosen as the system response. Based on preliminary information, it is known that the response surface is smooth and continuous. Hence, it may be

approximated by a lower-order polynomial. The design of experiment matrix with outcomes is given in Table 3.64 (trials 1 to 6).

The second-order regression model for hardness by Rockwell has this form:

$$\widehat{y}_1 = 22X_1 + 35X_2 + 51X_3 + 38X_1X_2 + 34X_1X_3 + 52X_2X_3 \tag{3.160}$$

Lack of fit of the obtained regression model has been checked in trials 7, 10, 11, 12 and 13. Control trials have been chosen under the assumption that they are in the optimum zone and may simultaneously be used for a model augmenting to a higher-order regression model. The analyzed regression model has not been adequate in control design points. To obtain an incomplete cube model, trial No. 7 has been used to calculate regression coefficients. The regression model of incomplete third order has this form:

$$\widehat{y}_2 = 22X_1 + 35X_2 + 51X_3 + 38X_1X_2 + 34X_1X_3 + 52X_2X_3 + 222X_1X_2X_3 \tag{3.161}$$

Lack of fit of the obtained model has been checked by trials 8, 9, 10, 14 and 15. Regression model (3.161) has not been adequate in chosen control trials. To calculate regression coefficients for a third-order model, it has been necessary to include these trials: 1, 2, 3 and 7, 8, 9, 10, 11, 12 and 13. The third-order regression model is:

$$\begin{aligned}\widehat{y}_3 = {} & 22X_1 + 35X_2 + 51X_3 + 11.25X_1X_2 + 38.25X_1X_3 + 58.50X_2X_3 \\ & +15.75X_1X_2(X_1 - X_2) + 51.75X_1X_3(X_1 - X_3) + 22.50X_2X_3(X_2 - X_3) \\ & +270X_1X_2X_3\end{aligned} \tag{3.162}$$

Lack of fit of the obtained regression model has been checked in control trials 4, 5, 6 and 14, 15, 16, 17 and 18. Three trials 16, 17 and 18 lie inside concentration triangle. The obtained third-order regression model has been adequate in all control trials. For a three-component mixture it is easiest to determine the optimum from geometric interpretation of the regression model. The contour lines of regression model (3.162) are shown in Fig. 3.41.

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Table 3.64 Design matrix for a three-component mixture

N	Design matrix			HRC	Augmenting of models		
	X_1	X_2	X_3		II order model	Incomplete III	III order
1	1	0	0	22	+	+	+
2	0	1	0	35	+	+	+
3	0	0	1	51	+	+	+
4	0.5	0.5	0	33	+	+	check of lack of fit
5	0.5	0	0.5	45	+	+	
6	0	0.5	0.5	56	+	+	
7	0.333	0.333	0.333	58	check of lack of fit	+	+
8	0.666	0.333	0	30		check of lack of fit	+
9	0.333	0.666	0	32			+
10	0	0.666	0.333	55	check of lack of fit		+
11	0	0.333	0.666	57			+
12	0.666	0	0.333	44			+
13	0.333	0	0.666	46			+
14	0.75	0.25	0	29		check of lack of fit	check of lack of fit
15	0.25	0.75	0	32			
16	0.50	0.25	0.25	54			
17	0.25	0.50	0.25	58			
18	0.25	0.25	0.50	56			

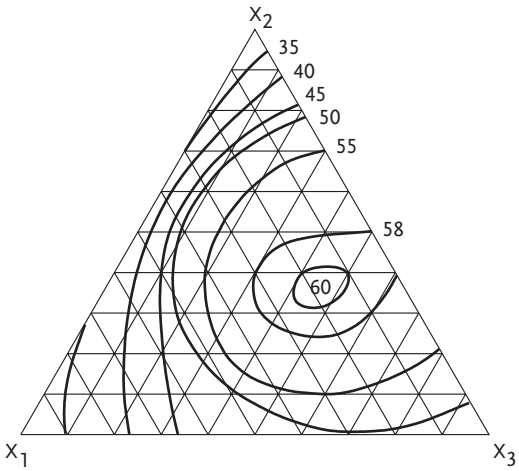

Figure 3.41 Contour lines of regression model Eq. (3.162)

The figure clearly shows that the optimum is in this factor space: Ni∈ (20–30 %); Cr∈ (30–50 %); B∈ (30–40 %).

It should be noted that by including trials No. 16, 17 and 18 one may calculate regression coefficients for a fourth order regression model.

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Appendix

A.1
Answers to Selected Problems

Chapter I

1.1

Sample	Values	Averages	Variances
1	3.4; 3.9; 3.9; 9.9; 3.5	5.7	8.4
2	5.5; 8.5; 6.7; 0.7; 4.5	5.2	4.8
3	1.5; 0.6; 3.3; 4.2; 5.7	3.6	4.9
4	2.2; 8.2; 9.4; 0.4; 9.9	4.9	12.3
5	8.4; 5.0; 0.8; 6.6; 3.1	4.1	9.2
6	9.9; 9.3; 5.1; 4.8; 9.2	5.9	10.5
7	6.5; 4.0; 9.2; 4.6; 2.1	3.9	7.4
8	1.8; 3.6; 5.2; 9.4; 2.9	4.9	8.9
9	7.5; 1.7; 5.6; 3.3; 8.0	4.5	7.1
10	4.2; 5.1; 2.3; 5.8; 2.6	3.8	4.8
11	6.8; 8.2; 3.6; 2.8; 8.3	5.4	6.9
12	6.1; 6.0; 2.8; 6.9; 9.1	4.8	12.1
13	3.5; 8.2; 6.5; 3.2; 2.9	4.5	6.5
14	3.6; 9.7; 8.6; 8.8; 2.3	6.0	6.2
15	5.2; 4.7; 8.8; 2.4; 7.4	5.1	5.2
16	0.9; 0.0; 4.7; 4.4; 5.9	4.2	11.9
17	7.5; 8.0; 6.8; 3.0; 0.0	3.7	12.2
18	6.8; 2.2; 0.2; 5.9; 7.0	4.1	10.9
19	9.4; 0.3; 4.0; 9.8; 0.9	4.6	15.1
20	8.6; 9.9; 1.4; 5.7; 6.4	5.9	6.3
	Averages:	4.7	8.5
	Population parameters:	4.5	8.3

Design of Experiments in Chemical Engineering. Živorad R. Lazić
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ISBN: 3-527-31142-4

1.2

a) P(Z≤1.4)-P(Z<00)=0.9192-0.5000=0.4192
b) P(Z<0)-P(Z<-0.78)=P(Z<0.78)-P(Z<0)=0.7823-0.500=0.2823
c) P(Z≤1.9)-P(Z≤-0.24)=0.9713-[1-P(Z≤0.24)]=0.9713-0.4052=0.5661
d) P(Z≤1.96)-P(Z≤0.75)=0.9750-0.7734=0.2016
e) P(-∞<Z≤0.44)=0.6700
f) P(-∞≤Z≤1.2)=0.8849
g) P(Z≤1)-P(Z≤-1)=0.8413-[1-P(Z≤1)]=0.8413-0.1587=0.6826
h) P(Z≤1.96)-P(Z≤-1.96)=0.975-[1-P(Z≤1.96)=0.975-0.025=0.9500
i) P(Z≤2.58)-P(Z≤-2.58)=0.9951-0.9949=0.9902

1.3

a) By Table B, Z_1 lies between 1.40 and 1.41 so that by linear interpolation we get Z_1=1.405.
b) Z_1=-1.3733.

1.4

$$Z = \frac{x-\mu}{\sigma} = \frac{26-20}{\sqrt{9}} = 2; P(Z \leq 2) = 0.97725$$

$$\Rightarrow P(Z \succ 2) = 1 - P(Z \leq 2) = 1 - 0.97725 = 0.02275$$

Hence, 2.28% of population may be expected to have values above 26.

1.5

a) 0.88; b) 0.128; c) 0.829;

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1.6

a) 0.4649; b) 0.2684; c) 0.0401; d) 0.2266;

1.7

a) 435; b) 92;

1.8

a) 90.5%; b) 0.0588;

1.9

a) n≈13; b) n=4; c) n=4;

1.10

$$\sigma_{\bar{x}} = \frac{\sigma}{\sqrt{n}} = \frac{7\times10^{-5}}{\sqrt{7}} = 2.646 \times 10^{-5};$$

$$Z_{1-\alpha/2} = Z_{0.975} = 1.96; \quad \bar{X} = 12.36 \times 10^{-5};$$

$$12.36 \times 10^{-5} - 1.96 \times 2.646 \times 10^{-5} \prec \mu \prec 12.36 \times 10^{-5} + 1.96 \times 2.646 \times 10^{-5}$$

$$7.17 \times 10^{-5} \prec \mu \prec 17.55 \times 10^{-5}$$

1.11

$\bar{X} = 65.5\left[BTU/HRFT^{2\,\circ}F\right]; S_X = 4.347\left[BTU/HRFT^{2\,\circ}F\right]; 1-\alpha = 0.99;$

α=0.01; $\alpha/2$=0.005;n=10-1=9; $t_{9;0.995}$=3.250

65.5-3.25 × 4.347/$\sqrt{10}$<K<65.5+3.25 × 4.347/$\sqrt{10}$

61.03<K<69.97

Minimal value with 99% confidence is: 61.03 [BTU/HR FT$^{2\,\circ}$K]

1.12

$\bar{X}_1$=15.770; $\bar{X}_2$=15.597; $Z_{1-\alpha/2}=Z_{0.975}$=1.96; $\sigma_1=\sigma_2$=0.016;

$$\bar{X}_1 - \bar{X}_2 + Z_{\alpha/2} \times \left(\frac{\sigma_1^2}{n_1} + \frac{\sigma_2^2}{n_2}\right)^{0.5} \prec \mu_1 - \mu_2 \prec \bar{X}_1 - \bar{X}_2 + Z_{1-\alpha/2} \times \left(\frac{\sigma_1^2}{n_1} + \frac{\sigma_2^2}{n_2}\right)^{0.5}$$

$$15.770\text{-}15.597\text{-}1.96\left(\frac{0.016}{3} + \frac{0.016}{3}\right)^{0.5} < \mu_1\text{-}\mu_2 < 15.770\text{-}15.597$$

$$+1.96\left(\frac{0.016}{3} + \frac{0.016}{3}\right)^{0.5}; -0.029 < \mu 1 - \mu 2 < 0.375$$

1.13

Replicated measurements will be treated as individual observations:

$\bar{X}_1 = 5.37;$ $\bar{X}_2 = 5.67;$ $S_1^2 = 0.08425;$ $S_2^2 = 0.07325.$

Assume that $\sigma_1^2 = \sigma_2^2$.

$$S_p^2 = \frac{(n_1-1)S_1^2+(n_2-1)S_2^2}{n_1+n_2-2} = \frac{2\times0.0845+2\times0.07325}{3+3-2} = 0.07875 \Rightarrow S_p = 0.28062$$

Since 1-α=0.95 and n_1=3; n_2=3, or f=n_1+n_2-2=4 we get from the table for t-distribution that: $t_{4;0.975}$=2.776.

$$5.37\text{-}5.67\text{-}2.776 \times 0.28062\sqrt{\frac{1}{3}+\frac{1}{3}} < \mu_1\text{-}\mu_2 < 5.37\text{-}5.67\text{+}2.776 \times 0.28062\sqrt{\frac{1}{3}+\frac{1}{3}}$$

$$-0.9361 < \mu_1 - \mu_2 < 0.2361$$

Since a 95% confidence interval has 0=μ_1-μ_2, which means that $\mu_1=\mu_2$, the catalysts do not differ in efficiency.

1.14

$\bar{X}$ =5.88; S_X^2 =0.00975; n=5; k=n-1=4; α=0.05;

$\chi^2_{4;0.025} = 0.484; \chi^2_{4;0.975} = 11.1$

$$\frac{4 \times 0.00975}{11.1} \le \sigma^2 \le \frac{4 \times 0.00975}{0.484}$$

$$0.00351 \le \sigma^2 \le 0.08057$$

1.15

$\bar{X}_1 = 310.8423\,[^{\circ}C]$; $S_1^2 = 1.1867$; $\bar{X}_2 = 310.5246\,[^{\circ}C]$; $S_1^2 = 1.5757$;

$k_1=n_1-1=12$; $k_2=n_2-1=12$; $\alpha=0.02$; $F_{12;12;0.01}=0.241$;

$F_{12;12;0.99}=4.16$.

$$\frac{1.1867}{1.5757}\times 0.241 \le \frac{\sigma_1^2}{\sigma_2^2} \le \frac{1.1867}{1.5757}\times 4.16$$

$$0.18104 \le \frac{\sigma_1^2}{\sigma_2^2} \le 3.13300$$

The associated 98% confidence interval for σ_1/σ_2 is:

$0.42548<\sigma_1/\sigma_2<1.7700$

Since the 98% confidence interval includes the value $1=\sigma_1/\sigma_2$ one may not state that there is a statistically significant difference between σ_1 and σ_2.

1.16

$\bar{X}=2.50\times 10^{11}$; $S^2=1.11\times 10^{20}$; $n=10$; $\alpha=0.10$; $t_{9;0.05}=-1.83$; $t_{9;0.95}=1.83$.

$$\bar{X}+t_{\alpha/2}\times\frac{S_X}{\sqrt{n}} \prec \mu \prec \bar{X}+t_{1-\alpha/2}\times\frac{S_X}{\sqrt{n}}$$

$2.50\times 10^{11}-1.83\times 1.05\times 10^{10}/\sqrt{n}<\mu<2.50\times 10^{11}+1.83\times 1.05\times 10^{10}/\sqrt{n}$

$2.446\times 10^{11}<\mu<2.556\times 10^{11}$

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1.17

$\bar{X}=11.411$; $S_X^2=3.5551$; $Z_{0.025}=-1.96$; $10.24<\bar{X}<12.58\ [mg/m^3]$

1.18

$$\sum X = 25.15;\ \sum X^2 = 22.0680;\ \bar{X} = \sum X/n = 0.838;$$

$$S_X^2 = \frac{n\sum X^2-(\sum X)^2}{n(n-1)} \Rightarrow S_X^2 = \frac{30\times 22.0680-25.15^2}{30(30-1)} = 0.03393;$$

$S_X=0.18420$; $f=n-1=29$; $\alpha=0.01$; $t_{29;0.005}=-2.76$; $t_{29;0.995}=2.76$

$$\bar{X}+t_{\alpha/2}\times\frac{S_X}{\sqrt{n}} \le \mu \le \bar{X}+t_{1-\alpha/2}\times\frac{S_X}{\sqrt{n}}$$

$0.838-2.76\times 0.18420/\sqrt{30}<\mu<0.838+2.76\times 0.18420/\sqrt{30}$

$0.745<\mu<0.931\ [\%Si]$

1.19

$\bar{X}_1$=8.57; S_1=17.95; n_1=7; $\bar{X}_2$=11.89; S_2^2=15.61; n_2=9;
f=14.46; $t_{f;1-\alpha/2}$=-1.601; $\bar{X}_1$-$\bar{X}_2$=2.073.

1.20

1. Assume normal distribution
2. H_0: $\mu \geq 26.5$; H_1: $\mu < 26.5$
3. Since σ^2 is unknown we use the next test statistic:

$$T = \frac{\bar{X} - \mu}{S/\sqrt{n}}$$

4. Let α=0.05;
5. T-has Student's distribution with f=n-1=11-1=10 degrees of freedom.
6. $\bar{X}$=26.03568; $S/\sqrt{11}=6.741 \times 10^{-5}$

$$T = \frac{26.03568 - 26.5}{6.741 \times 10^{-5}} = -\frac{0.46432}{6.741} \times 10^5 = -6.888 \times 10^3$$

7. $t_{10;0.95}$=1.81 from Table C; Hence:
T=$-6.888 \times 10^3 < t_{10;0.95}$=1.81 so that H_0 is rejected or we may say that μ does not exceed 26.5. A chance to make an error in this statement is 5 to 100 or α=0.05.

1.21

$\bar{X}$=54.76; S^2=4.216; S=2.053; α=0.01; f=n-1=49

$$T = \frac{54.76 - 55.0}{2.053/\sqrt{50}} = -0.93; t_{49;0.01} = -2.704$$

H_0 with 99% confidence is not rejected.

1.22

1. Assume a normal distribution
2. H_0: $\mu_1 = \mu_2$; H_1: $\mu_1 \neq \mu_2$
3. Since we know population variance $\sigma_1^2 = \sigma_2^2$ =0.016 the test statistic is:

$$Z = \frac{\bar{X}_1 - \bar{X}_2 - \left(\mu_1 - \mu_2\right)}{\sigma \times \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} = \frac{15.770 - 15.597 - \left(\mu_1 - \mu_2\right)}{\sqrt{0.016}\sqrt{\frac{1}{3} + \frac{1}{3}}} = 1.675$$

4. α=0.05
5. Z has normal distribution.
6. Z=1.675
7. $Z_{1-\alpha/2}$=1.96; $Z_{\alpha/2}$=-1.96 since: -1.96<1.675<1.96 the null hypothesis $\mu_1 = \mu_2$ is accepted, or both bottles are with the same HCl.

1.23

$\bar{X}_1$=14.280; S_1^2=0.028; n_1=4; $\bar{X}_2$=14.374; S_2^2=0.004; n_2=3

$$T = \frac{\bar{X}_1 - \bar{X}_2}{\left(\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}\right)^{0.5}} = \frac{14.374 - 14.280}{\left(\frac{0.028}{4} + \frac{0.004}{3}\right)^{0.5}}$$

T=1.0330;

$$f = \frac{\left(\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}\right)^2}{\frac{\left(S_1^2/n_1\right)^2}{n_1+1} + \frac{\left(S_2^2/n_2\right)^2}{n_2+1}} - 2$$

$$f = \frac{6.944 \times 10^{-5}}{9.8000 \times 10^{-6} + 4.4444 \times 10^{-7}} - 2 = 4.77 = 5$$

α=0.05; $t_{5;0.05}$=2.01; T=1.0330<$t_{5;0.05}$=2.01

The means of burning rates are not statistically different with 95% confidence.

1.24

S_1^2=0.028; n_1=4; S_2^2=0.004; n_2=3; k_1=3; k_2=2

$$F_R = \frac{S_1^2}{S_2^2} = \frac{0.028}{0.004} = 7.00;$$

$$F_{0.05}(3.2) = \frac{1}{F_{0.95}(3.2)} = \frac{1}{19.2} = 0.05$$

$$0.05 \prec F_R = 7.00 \prec 19.2$$

We accept H_0 with 95% confidence that there exist statistically significant differences between variances of these two samples.

1.25

$\bar{X}_1$=14.280; S_1^2=0.0280; n_1=4; $\bar{X}_2$=15.681; S_2^2=0.0024; n_2=4

$$T = \frac{\bar{X}_1 - \bar{X}_2}{\left(\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}\right)^{0.5}} = \frac{15.681 - 14.280}{\left(\frac{0.028}{4} + \frac{0.0024}{4}\right)^{0.5}}$$

T=16.0665;

$$f = \frac{\left(\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}\right)^2}{\frac{\left(S_1^2/n_1\right)^2}{n_1+1} + \frac{\left(S_2^2/n_2\right)^2}{n_2+1}} - 2$$

$$f = \frac{5.8 \times 10^{-5}}{1.0 \times 10^{-5}} - 2 = 5.8 - 2 = 3.8 \approx 4;$$

α=0.05; $t_{4;0.05}$=2.13 T=16.0665>$t_{4;0.05}$=2.3

DropBooks

It is asserted with 95% confidence that there are statistically significant differences in effects of the two catalyst types on burning rate.

1.26

Source of variations	f	SS	MS	F	$F_{1;8;0.95}$
Factor A	1	0.146	0.146	5.84	5.32
Factor B	1	0.002	0.002	0.08	5.32
Factor C	1	0.300	0.300	12.00	5.32
Interaction AB	1	0.000	0.000	0.00	5.32
Interaction AC	1	0.104	0.104	4.16	5.32
Interaction BC	1	0.253	0.253	10.12	5.32
Interaction ABC	1	0.041	0.041	1.64	5.32
Error	8	0.200	0.025	–	–
Total	15	1.046	–	–	–

1.27

a) $S_X^2 = 24.596$; $S_p^2 = 1.808$; $F = 13.6 \succ F_{3;20;0.95} = 3.10$

b) $t_{20;0.95}$=2.086; $36.522 < \mu_{III} < 38.812$

1.28

$S_X^2 = 5.1566$; $S_p^2 = 0.89391$; $F = 5.77 \succ F_{3;36;0.95} = 2.85$

1.29

$S_X^2 = 0.0014167$; $S_p^2 = 0.0007458$; $F = 1.899 \prec F_{3;12;0.99} = 5.95$

$S_p = 0.01362$; $t_{12;0.995} = 3.055$; $\mu_{\bullet 1} = 0.385 \pm 3.055 \times 0.01362$

1.30

SS_C=5,653,437.5; SS_R=170,343,939.6; SS_E=7,306,879.2;

SS_T=310,933,275; pressure does not effect the moment but it does the actuator model.

1.31

Source of variation	f	SS	MS	F	$F_{1;12;0.95}$
Factor methods	1	0.00001	0.00001	34.61335	4.75
Factor mixing	1	0.00003	0.00003	112.23636	4.75
Interaction	1	0.00000	0.00000	0.19043	4.75
Error	12	0.00000	0.00000	–	–
Total	15	0.00004	–	–	–

1.32

Source of variation	F	SS	MS	F	$F_{1;24;0.95}$
Cutting tools (T)	1	2.82	2.82	1.21	4.26
Cutting angle (B)	1	20.32	20.32	8.72	4.26
Type of cutting (C)	1	31.01	31.01	13.31	4.26
Interaction T × B	1	0.20	0.20	0.09	4.26
Interaction T × C	1	0.01	0.01	0.004	4.26
Interaction B × C	1	0.94	0.94	0.40	4.26
Interaction T × B × C	1	0.19	0.19	0.08	4.26
Error	24	53.44	2.33	–	–
Total	31	108.93	–	–	–

DropBooks

1.33

Source of variation	f	SS	MS	F	F_{TAB}
Samples	11	93.486	8.499	7.691	$F_{11;22;0.95}$=2.26
Kivets	2	14.527	7.263	6.573	$F_{2;22;0.95}$=3.44
Bottles	1	0.347	0.347	0.314	$F_{1;22;0.95}$=4.30
Samples × kivets	22	22.806	1.037	0.938	$F_{22;22;0.95}$=2.05
Samples × bottles	11	27.153	2.468	2.233	$F_{11;22;0.95}$=2.26
Kivets × bottles	2	1.695	0.847	0.767	$F_{2;22;0.95}$=3.44
Samples × kivets × bottles	22	24.305	1.105*	–	–
Total	71	184.319	–	–	–

* Three-factor interaction variance is used for estimation of residual variance

1.34

The experimental program has been done with no trial replication, so that residual variance must be determined based on variance interactions. Whether one may neglect all interactions or some of the variance interactions may be included in the residual variance since its effect is significant and different from others, is checked against *Bartlett's* criterion. Assuming that variance interactions are negligible, we have:

Source of variation	f	SS	MS	F	$F_{1;4;0.95}$
Factor A	1	3.125	3.125	1.87	7.71
Factor B	1	47.045	47.045	28.17	7.71
Factor C	1	0.720	0.720	0.43	7.71
Interaction AB	1	0.720	0.720	–	–
Interaction AC	1	0.045	0.045	–	–
Interaction BC	1	0.405	0.405 1.670	–	–
Interaction ABC	1	0.500	0.500	–	–
Total	7	52.560		–	–

1.35

Source of variation	f	SS	MS	F	F_{TAB}
Between batches	8	792.88	99.11	132.15	$F_{8;27;0.95}$=2.31
Between samples	2	4.21	2.11	2.81	$F_{2;27;0.95}$=3.35
Interaction batches × samples	16	21.09	1.32	1.76	$F_{16;27;0.95}$=2.02
Error	27	20.17	0.75	–	–
Total	53	838.35	–	–	–

1.36

Source of variation	f	SS	MS	F	F_{TAB}
Between chalks	1	0.503040	0.503040	62880.00	$F_{1;44;0.95}$=4.04
Between laboratories	10	0.005223	0.000522	65.25	$F_{10;44;0.95}$=2.03
Interaction laboratory × chalks	10	0.000478	0.000048	6.00	$F_{10;44;0.95}$=2.03
Error	44	0.000363	0.000008	–	–
Total	65	0.509104	–	–	–

1.37

Source of variation	f	SS	MS	F	F_{TAB}
Factor A	4	478 463	119 615.75	373.53	$F_{4;24;0.95}$=2.78
Factor B	2	52 794	26 397.00	82.41	$F_{2;24;0.95}$=3.40
Factor C	3	150 239	50 079.67	156.34	$F_{3;24;0.95}$=3.01
Interaction AB	8	16 807	2 100.88	6.56	$F_{8;24;0.95}$=2.36
Interaction AC	12	53 890	4 490.83	14.02	$F_{12;24;0.95}$=2.18
Interaction BC	6	6 416	1 069.33	3.34	$F_{6;24;0.95}$=2.51
Interaction ABC	24	7 688	320.33		
Total	59	766 297	–	–	–

Since experiments have been done with no replications, the residual variance has been determined based on the variance interaction ABC, as it has been proved by theoretical analysis that it may be neglected.

1.38

Source of variation	f	SS	MS	F	F_{TAB}
Between locations	3	128.74	42.91	13.41	$F_{3;105;0.95}$=2.70
Between alloys	8	128.18	16.02	5.01	$F_{8;105;0.95}$=2.04
Between researchers	3	106.24	35.41	11.07	$F_{3;105;0.95}$=2.70
Location × alloy	24	89.32	3.72	1.16	$F_{24;105;0.95}$=1.65
Location × researcher	9	12.73	1.41	–	–
Researcher × alloy	24	23.32	0.97 3.20	–	–
Location × alloy × researcher	72	58.96	0.82	–	–
Total	143	547.49	–	–	–

Since an analysis of the three-factor variance is with no replications, for an estimate of residual variance one may pool variances of the three last interactions, as they cannot be significant theoretically.

1.39

F_{flow}=165.707; $F_{conc.}$=7.607; MS_E=1.90276 (pooled AB error).

At 95% confidence, catalyst flow and concentration have a statistically significant effect on conversion of vinegar acid.

1.40

DropBooks

SS_C=39,934.1875; SS_R=324,082.1875; SS_E=9,232.0625
MS_C=13,311.3958; MS_R=108,027.3958; MS_E=1025.7847;
F_C=12.97679>$F_{3;9;0.99}$=6.99;F_R=105.31196>$F_{3;9;0.99}$=6.99;

Interaction AB pooled in residual variance with error variance; it is not extracted as there have been no trials replications.

1.41

$SS_{ADH.}$=6274.307; $SS_{PRA.}$=4.380; effect of a preliminary surface preparation is insignificant, while the effect of adhesion systems is significant.

$F_{ADH.}$=150.422; $F_{PRA.}$=0.315; $F_{ADH.\times PRA.}$=19.304;
$F_{1;40;0.95}$=4.08; $F_{3;40;0.95}$=2.84.

1.42

$SS_{PRA.}$=9.275; $SS_{ADH.}$=13,761.231; $SS_{PRA.\times ADH.}$=266.21.

All effects except surface preparation with a primer are significant with 95% confidence.

$F_{PRA.}$=0.83442; $F_{ADH.}$=412.66618; $F_{PRA.\times ADH.}$=7.98297;
$F_{1;40;0.95}$=4.08; $F_{3;40;0.95}$=2.84.

1.43

Source of variation	f	SS	MS	F	F_{TAB}
Adhesive × (A)	3	17 721.30	5 907.10	472.20	$F_{3;80;0.95}$=2.70
Preparation × (B)	1	13.20	13.20	1.06	$F_{1;80;0.95}$=3.96
Thickness × (C)	1	4 256.01	4 256.01	340.22	$F_{1;80;0.95}$=3.96
Interaction A × B	3	951.79	317.26	25.36	$F_{3;80;0.95}$=2.70
Interaction A × C	3	2 314.24	771.41	61.67	$F_{3;80;0.95}$=2.70
Interaction B × C	1	0.45	0.45	0.04	$F_{1;80;0.95}$=3.96
Interaction A × B × C	3	119.64	39.88	3.19	$F_{3;80;0.95}$=2.70
Error	80	1 000.78	12.51	–	–
Total	95	26 377.41	–	–	–

1.44

Source of variation	f	SS	MS	F	$F_{1;8;0.95}$
Type of catalyst	1	5.752	5.752	410.857	5.32
Batch of AP	1	0.024	0.024	1.714	5.32
Interaction	1	0.023	0.023	1.643	5.32
Error	8	0.110	0.014	–	–
Total	11	5.909	–	–	–

1.45

$X_{EKS.} = 5.2; \quad \bar{X} = 4.39; \quad S_X^2 = 0.0481; \quad S_X = 0.2193; \quad N = 13; \quad f = 12;$

$\Delta X_{MAX} = 5.2 - 4.39 = 0.81; \quad f_0 = 0; \quad t_{\alpha=0.05}^{f_0+f-1} = t_0^{11} = 1.80;$

$$\left[\frac{13C^2 \times 11}{12\left(12 - \frac{13C^2}{12}\right)}\right]^{0.5} = 1.80; \quad C \approx 1.59$$

$\Delta X_{MAX} = C \times S_X = 1.59 \times 0.2193 = 0.35$

$\Delta X_{MAX} = 0.81 \succ 0.35; X = 5.2$ outlier

1.48

$X_{EKS.} = 14.531; \bar{X} = 14.196; S_X^2 = 9.350 \times 10^{-6}; S_X = 0.003; N = 4; f = N - 1 = 3;$

$\Delta X_{MAX} = X_{MAX} - \bar{X} = 14.531 - 14.196 = 0.335; f_0 = 0; t_{\alpha=0.05}^{f_0+f-1} = t_{0.05}^2 = 2.92;$

$$\left[\frac{NC^2(f+f_0-1)}{f\left(f+f_0-\frac{NC^2}{f}\right)}\right]^{0.5} = 2.92; \left[\frac{4C^2\times 2}{3\left(3-\frac{4C^2}{3}\right)}\right]^{0.5} = 2.92 \Rightarrow C = 1.35;$$

$\Delta X_{MAX} = C \times S_X = 1.35 \times 0.003 = 0.004$

$\Delta X_{MAX} = 0.335 \succ 0.004;\ X = 14.531$ outlier

1.49

a) b_0=9.27; b_1=1.44; $\widehat{Y}$=9.27+1.44X

b)

Source of variation	f	SS	MS	F	$F_{1;9;0.95}$
Regression	1	226.95	226.95	96.17	5.12
Residual	9	21.23	2.36	–	–
Corrected total	10	248.18	–	–	–

c) $1.11 \leq \beta_1 \leq 1.77$

d) $12.15 \leq \mu \leq 15.03$ (X=3)

e) $12.15 \leq \mu \leq 15.03$ (X=3) $6.61 \leq \mu \leq 23.89$(X=-2)

DropBooks

1.50

Source of variation	f	SS	MS	F	$F_{5;6;0.95}$
Regression	1	3 293.77	3 293.77	–	–
Residual	11	102.85	9.35	–	–
Lack of fit	5	91.08	18.22	9.30*	4.39
Pure error	6	11.77	1.96	–	–
Total	12	3 396.62	–	–	–

$\widehat{Y}$=129.7872-24.0199X

1.51

$\bar{X} = 6668.4; \quad \bar{Y} = 8662.6; \quad \widehat{Y} = 1360.0 + 1.0951X$

1.52

$\bar{X} = 751.875; \quad \bar{Y} = 316.75; \quad \widehat{Y} = 4654.9846 - 0.4498X$

1.53

$\bar{X} = 2165.000; \quad \bar{Y} = 2125.278; \quad \widehat{Y} = 15.352 + 0.974561X$

Source of variation	f	SS	MS	F	$F_{1;16;0.95}$
Regression	1	4 772 451.7	4 772 451.7	1 501.01	4.49
Residual	16	50 871.9	3 179.49	–	–
Total	17	4 823 323.6	–	–	–

1.54

Source of variation	f	SS	MS	F	$F_{4;6;0.95}$
Regression	1	52.50	52.50	–	–
Residual	10	17.17	1.717	–	–
Lack of fit	4	5.50	1.375	0.706	4.53
Pure error	6	11.67	1.945	–	–
Total	11	69.67	–	–	–

a) $\widehat{Y}$ = -21.33+5.0X b) $2.984 \leq \mu_1 \leq 7.016$

1.55

b_0=0.353332; $b_1=3.34292 \times 10^{-4}$

$\widehat{Y}= 0.353332+3.34292 \times 10^{-4} \times t$

1.56

$\widehat{Y}$=45.1972-2.68408X_1+4.20910X_2; r^2=0.94808

1.57

a) $\widehat{X}$=14.7075-1.2042Z_1-0.46288Z_2; b) r^2=0.82523

1.58

$\widehat{Y}_1$=3.09204+0.193694X+0.0362872X^2; r^2=0.99956;
$\widehat{Y}_2$=2.41588+0.180067X+0.034122X^2; r^2=0.998707;

1.59

$\widehat{Y}$=3.73926+1.7717X-0.0601562X^2; r^2=0.991516

1.60

a) $\widehat{Y}$=2.5372000-0.0004718X b) Regression model is adequate

Source of variation	f	SS	MS	F	F_{TAB}
Regression	1	0.110395	0.110395	14.50	$F_{1;13;0.95}$=4.67
Residual	13	0.098938	0.007611	–	–
Lack of fit	5	0.018938	0.003788	0.38	$F_{5;8;0.95}$=3.69
Pure error	8	0.080000	0.010000	–	–
Total	14	0.209333	–	–	–

c) 95 % confidence interval of Y for:

X=0; X=$\bar{X}$; X=400; X=460.

1. X=0 μ±2.160 × 0.527=μ±1.138
2. X=$\bar{X}$ μ±2.160 × 0.022=μ±0.048
3. X=400 μ±2.160 × 0.039=μ±0.084
4. X=460 μ±2.160 × 0.048=μ±0.104.

Chapter II

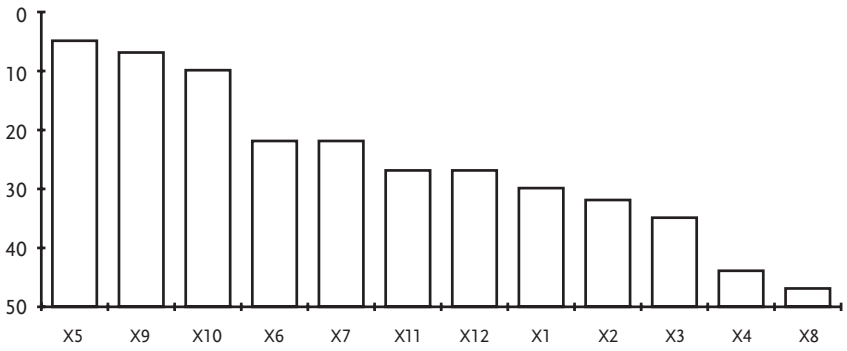
2.1

Based on Table 2.14:

$$\sum_{1}^{4} T_j = 156; \quad T = 26; \quad S = 1650.$$

$$\omega = \frac{12 \times 1650}{16(1728 - 12) - 4 \times 156} = 0.738;$$

From Table D for α=0.05 and f=12-1=11 we get χ^2_T=19.75. The null hypothesis in accord of researchers' opinions is accepted. Histogram of rank sum is given in the figure.



Histogram of ranks

DropBooks

It is evident from the diagram that we have here an even nonmonotonous curve of ranks, so the suggestion here is to take the eight most significant factors as basis of the next phase of experimental studies.

2.2

$\omega = 0.526$

2.3

$$\omega = \frac{12\times 14665}{18^2\left(11^3-11\right)-18\times 946} = 0.43;\ \chi_R^2 = 18(11-1)\times 0.43 = 77.4;$$
$$\chi^2_{T(0.01;10)} = 23.2$$

2.4

$\omega = 0.012;\quad \chi_R^2 = 4.9.$

2.5

Screened factors are X_1;X_2;X_3;X_4;X_5 and X_6.

The regression has this form:

$\widehat{Y}$=67.4+10.7X_1+3.9X_2-7.0X_3+3.45X_4+1.2X_5+3.8X_6

Estimated response values are by regression given in Table 2.40.

2.6

$E_{X_1} = -3.05;$	$E_{X_2} = 64.90;$		first screening
$E_{X_1X_2} = -13.30;$	$E_{X_7} = -0.97;$	$E_{X_8} = 3.12;$	second screening;
$E_{X_2X_3} = -5.02;$	$E_{X_8} = 4.72;$	$E_{X_3} = 4.25;$	third screening;
$E_{X_3X_4} = -2.75;$	$E_{X_5} = -1.25;$		fourth screening;
$E_{X_1X_3} = 2.00;$	$E_{X_4} = 1.00;$		fifth screening.

Finally, we screened out factors X_1;X_2 and X_3. Factors X_4 and X_8 are qualitative with two levels.

2.7

SS_C = (275625.0+240100.0+334084.0+295936.0)/(3 × 3)-4566769.0/(3 × 3 × 4)⇒
SS_C = 127305.00-126854.69=450.31
SS_R = (355216.0+528529.0+662596.0)/(4 × 3)-126854.69⇒
SS_R = 128861.75-126854.69=2007.06
SS_{CR} = (23104+18496+25281+22201+31684+27225+37636+36100+
+ 38025+35721+50625+42025)/3-127305.0-128861.75+126854.69
SS_{CR} = 62.27; SS_E=129995.00-126854.69=3140.31

Source of variation	f	SS	MS	F	F_{TAB}
Between blocks	3	450.31	150.10	14.46	$F_{3;6;0.95}$=4.76
Between levels of factors	2	2 007.06	1 003.53	96.68	$F_{2;6;0.95}$=5.14
Experimental error	6	62.27	10.38	–	–
Sampling error	24	3 140.31	130.85	–	–
Total	35	5 659.95	–	–	–

2.8

SS_C = (30625.00+26676.69+37121.73+32880.57)/3-(507414.03)/(3 × 4)
SS_C = 42434.66-42284.50=150.16
SS_R = (39469.77+58723.83+73619.97)/4-42284.50
SS_R = 668.89
SS_E = 43124.30-42284.50-150.16-668.89=20.75

Source of variation	f	SS	MS	F	F_{TAB}
Between blocks	3	150.16	50.05	14.47	$F_{3;6;0.95}$=4.76
Between levels of factors	2	668.89	334.45	96.66	$F_{2;6;0.95}$=5.14
Experimental error	6	20.75	3.46	–	–
Total	11	839.80	–	–	–

2.9

DropBooks

Source of variation	f	SS	MS	F	F_{TAB}
Type of mixing	6	6 317	1 053	2.33	$F_{6;30;0.90}$=1.98
Active matter	6	99 157	16 526	36.64	$F_{6;30;0.99}$=3.17
Dissolvent type	6	5 487	915	2.02	$F_{6;30;0.90}$=1.98
Experimental error	30	13 541	451	–	–
Total	48	124 502	–	–	–

2.10

The missing data is estimated and it is 90. Analysis of variance offered these results:

Source of variation	f	SS	MS	F	F_{TAB}
Nozzles in operation	3	379.19	126.397	2.250	–
Air flow	3	86.19	28.73	0.511	–
Water pressure	3	101.19	33.73 (32.17)**	0.573	–
Experimental error	5*	280.87	56.174	–	–
Total	15*	847.44	–	–	–

* One degree of freedom less because of estimation one data
** Corrected because of estimation

No factor is significant for the efficiency of scrubber, which may be explained either that real factors have not been included in the research or that variation intervals of analyzed factors are too small.

2.11

Source of variation	f	SS	MS	F	$F_{4;12;0.95}$
Stators	4	1 302.8	325.7	27.07	3.26
Rotors	4	36.4	9.10	0.76	3.26
Quality of insulation	4	52.4	13.10	1.09	3.26
Experimental error	12	144.4	12.03	–	–
Total	24	1 536.0	–	–	–

2.12

$$SS_R = \left(22^2 + 18^2 + 21^2 + 19^2\right)/4 - 80^2/16 = 2.50$$

$$SS_C = \left(18^2 + 17^2 + 26^2 + 19^2\right)/4 - 80^2/16 = 12.50$$

$$SS_L = \left(26^2 + 21^2 + 17^2 + 16^2\right)/4 - 80^2/16 = 15.5$$

$$SS_G = \left(23^2 + 20^2 + 17^2 + 20^2\right)/4 - 80^2/16 = 4.5$$

$$SS_E = 446 - 400 - 2.50 - 12.50 - 15.5 - 4.5 = 11.00$$

$$SS_T = 446 - 400 = 46.00$$

Source of variation	f	SS	MS	F	$F_{3;3;0.95}$
Between rows	3	2.50	0.83	0.23	9.28
Between columns	3	12.50	4.17	1.14	9.28
Between latin letters	3	15.50	5.17	1.41	9.28
Between numbers	3	4.50	1.50	0.41	9.28
Experimental error	3	11.00	3.67	–	–
Total	15	46.00	–	–	–

2.13

Source of variation	f	SS	MS	F	$F_{4;8;0.95}$
Temperature	4	15 100.56	3 775.14	26.29	21.4
Time	4	3 195.36	798.84	5.56	21.4
Reactor	4	200.76	50.19	0.35	21.4
Operator	4	254.16	63.54	0.44	21.4
Experimental error	8	1 148.92	143.62	–	–
Total	24	19 899.76	–	–	–

2.14

Source of variation	f	SS	MS	F	F_{TAB}
Cycles	–	288.92	–	–	–
Corrected-cycles	3	147.25	49.08	45.44	$F_{3;3;0.95}$=9.28
Corrected-dyestuff	3	164.58	54.86	50.80	$F_{3;3;0.95}$=9.28
Positions	2	8.17	4.09	3.79	$F_{2;3;0.95}$=9.55
Residual	3	3.25	1.08	–	–
Total	11	464.92	–	–	–

2.15

Source of variation	f	SS	MS	F	F_{TAB}
Corrected conditions	6	0.57	0.10	1.25	$F_{6;12;0.95}$=3.0
Corrected producer	6	1.06	0.18	2.25	$F_{6;12;0.95}$=3.0
Producer	–	0.99	–	–	–
Type of saw	3	0.04	0.01	0.13	$F_{3;12;0.95}$=3.49
Residual	12	0.96	0.08	–	–
Total	27	2.56	–	–	–

$$SS_1 = \left(7.50^2 + 8.14^2 + 7.26^2 + 6.77^2 + 8.08^2 + 6.84^2 + 9.03^2\right)/$$

$$4 - 53.62/(7 \times 4) = 0.99$$

$$SS_2 = \frac{7-1}{7\times 4^2(4-1)}\left[(4 \times 7.52 - 7.50 - 8.14 - 7.26 - 8.08)^2 + \ldots\right.$$

$$\left.\ldots + (4 \times 8.69 - 7.50 - 8.14 - 6.77 - 9.03)^2\right] = 0.57$$

$$SS_3 = \left(7.52^2 + 7.24^2 + 7.99^2 + 7.41^2 + 6.94^2 + 7.83^2 + 8.69^2\right)/4 - 102.68 = 0.50$$

$$SS_4 = \frac{7-1}{7\times 4^2(4-1)}\left[(4 \times 7.50 - 7.52 - 7.41 - 7.83 - 8.69)^2 + \ldots\right.$$

$$\left.\ldots + (4 \times 9.03 - 7.99 - 6.94 - 7.83 - 8.69)^2\right] = 1.06$$

$$SS_5 = \left[(2.30 + 1.64 + 1.92 + 2.14 + 1.65 + 1.64 + 1.86)^2 + \ldots\right.$$

$$\left.\ldots + (1.62 + 1.93 + 2.22 + 1.58 + 1.61 + 1.74 + 2.67)^2\right]/7 - 102.68 = 0.04$$

$$SS_T = \left(1.62^2 + 2.10^2 + 1.50^2 + 2.30^2 + \ldots + 1.86^2 + 2.62^2\right) - 102.68 = 2.56$$

$$SS_E = 2.56 - 0.99 - 0.57 - 0.04 = 0.96$$

DropBooks

2.16

$b_0 = 22.48 \times 10^{-3}$; $b_1 = -1.01 \times 10^{-3}$; $b_2 = 19.46 \times 10^{-3}$
$b_3 = 1.45 \times 10^{-3}$; $b_4 = -1.05 \times 10^{-3}$; $b_{12} = 2.24 \times 10^{-3}$;
$b_{13} = -1.32 \times 10^{-3}$; $b_{14} = 1.80 \times 10^{-3}$.

2.17

$$\gamma_a = 1.94 + 0.17X_1 - 0.39X_2 - 0.32X_3 + 0.10X_4 - 0.08X_1X_3 - 0.09X_2X_3 + 0.09X_2X_4 + 0.07X_3X_5$$

$$\gamma_b = 0.54 + 0.12X_2 + 0.16X_3 - 0.09X_4 - 0.18X_5 + 0.07X_1X_3 - 0.06X_1X_4 - 0.06X_1X_5 + 0.095X_2X_3 - 0.08X_2X_4 - 0.06X_3X_4 - 0.11X_3X_5 + 0.05X_4X_5$$

2.18

$$\widehat{y} = 47.1 + 2.78X_1 - 1.29X_2 + 3.23X_3 + 1.55X_4 + 1.35X_5 - 4.9X_6$$

2.19

$$\widehat{y} = 4.5250 + 0.1312X_1 - 0.0187X_2 + 0.4812X_3 - 0.0937X_4 + 0.0625X_1X_2 - 0.0625X_1X_3 - 0.05X_2X_3$$

2.20

y	Regression coefficients															
	b_0	b_1	b_2	b_3	b_4	b_5	b_6	b_7	b_{12}	b_{13}	b_{14}	b_{15}	b_{16}	b_{17}	b_{27}	b_{127}
y_1	59.1	-3.4	-7.4	0.0	-4.2	1.7	3.1	1.0	1.1	0.6	-1.9	-2.2	-1.2	-0.4	-0.4	-0.5
y_2	8.11	-2.19	-3.77	1.07	-1.54	2.78	-0.25	-0.42	0.34	1.16	-0.55	-1.47	-0.87	0.82	-0.16	-0.34
y_3	46.3	-8.1	-14.1	4.0	-6.0	10.1	-2.0	-1.8	0.6	4.5	-1.9	-5.1	-2.2	2.9	-0.5	-1.0
y_4	115	-6	-11	-7	-6	10	-6	-2	-10	2	1	0	7	-5	-3	1
y_5	31.2	-13.8	-22.5	8.6	-12.7	0	0.6	1.6	7.9	6.0	-4.7	2.2	-1.4	-0.8	0.3	-2.4
y_6	7.9	-0.7	0.0	0.0	-0.4	0.2	0.1	0.7	-0.4	-0.1	0.1	0.2	0.3	-0.1	0.4	0.2
y_7	109	2	-1	6	0.0	-2	0	5	-5	0	2	-4	-1	3	4	0
y_8	9.73	-0.12	-0.08	-0.07	0.36	-0.27	0.07	0.48	-0.22	-0.04	-0.14	0.43	0.10	0.06	-0.07	0.56
y_9	1.09	-0.17	0.25	-0.12	0.30	0.18	-0.19	0.13	-0.19	0.17	0.01	-0.14	0.12	-0.20	0.20	0.05
y_{10}	16.4	5.9	-3.2	-2.8	0.0	2.3	-0.1	-0.8	0.7	-2.0	0.3	-0.7	-1.2	0.3	1.1	1.2
y_{11}	1.096	0.016	0.001	-0.031	-0.001	0.002	0.002	0.002	0.001	-0.007	0.002	-0.001	0.001	0.003	0.002	-0.001

2.21

$$\widehat{y} = -4.2482 + 3.60625X_1 + 0.77565X_2 + 0.7814X_1^2 + 0.0588X_2^2 + 0.00072498X_1X_2$$

2.22

$$\widehat{y} = 0.2993 - 0.0829X_1 + 0.0738X_2 - 0.149X_3$$

$b_0 - y_0$=0.2993-(0.1224+0.1382+0.1204+0.0943+0.0698+0.1135)/6 =0.1896

$$\widehat{y} = 0.10975 + 0.0816X_1 + 0.0753X_2 - 0.1483X_3 - 0.6043X_1X_2 + 0.0345X_1X_3 - 0.0229X_2X_3 + 0.0359X_1^2 + 0.0321X_2^2 + 0.1224X_3^2$$

2.23

$$\widehat{y} = 353.00 - 145.07X_1 - 192.77X_2 + 133.70X_3 + 6.00X_1X_2 - 41.13X_1X_3 - 52.25X_2X_3 + 41.76X_1^2 + 51.35X_2^2 - 2.65X_3^2$$

2.24

$b_0 = 0.22006$; $b_1 = -0.0195$; $b_2 = 0.002169$; $b_3 = 0.025928$;
$b_4 = -0.10837$; $b_5 = -0.00446$; $b_{11} = 0.04943$; $b_{12} = -0.00375$;
$b_{13} = -0.056249$; $b_{14} = 0.3387$; $b_{15} = -0.04199$; $b_{22} = 0.03688$;
$b_{23} = 0.00399$; $b_{24} = 0.008625$; $b_{25} = -0.010499$; $b_{33} = 0.0296$;
$b_{34} = -0.0165$; $b_{35} = 0.071625$; $b_{44} = 0.0340$; $b_{45} = 0.018249$;
$b_{55} = 0.06427$.

2.25

$b_0 = 4.114$; $b_1 = 0.508$; $b_2 = 1.021$; $b_3 = -1.192$; $b_4 = -1.625$;
$b_{11} = 0.043$; $b_{12} = -0.212$; $b_{13} = -0.025$; $b_{14} = -0.719$; $b_{22} = 0.249$;
$b_{23} = 0.212$; $b_{24} = 0.245$; $b_{33} = 0.718$; $b_{34} = 0.212$; $b_{44} = 0.456$.

DropBooks

y=H/H_0

H_0-height of non-fluidized bed;

H- height of fluidized bed.

2.26

$$\widehat{y} = 93 + 20X_1 - 30X_2 - 58X_3 + 9X_1X_2 + 11X_2X_3 - 22X_1X_3$$

$$\widehat{y} = 50 + 16X_1 - 30X_2 - 63X_3 + 11X_2^2 + 29X_3^2 + 9X_1X_2 + 11X_2X_3 - 22X_1X_3$$

2.27

y	b_0	b_1	b_2	b_3	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
y_1	139.12	16.49	17.88	10.91	-4.01	-3.45	-1.57	5.13	7.13	7.88
y_2	1 261.11	268.15	246.50	139.48	-83.55	-124.79	199.17	69.38	94.13	104.38
y_3	400.38	-99.67	-31.40	-73.92	7.93	17.31	0.43	8.75	6.25	1.25
y_4	68.91	-1.41	4.32	1.63	1.56	0.06	-0.32	-1.63	0.13	-0.25

2.28

Name	Factors				Response					
	X_1	X_2	X_3	X_4	y_{u1}	y_{u2}	y_{u3}	y_{u4}	$\bar{y}$	$\hat{y}$
Basic level	1.5	4.25	54.0	9.5						
Variation intervals	1.0	0.5	12.0	5.0						
Regression coefficients b_i	0.131	-0.019	0.481	-0.094						
Product $b_i \times \varepsilon_i$	0.131	–	5.780	–						
Basic step h_a	–	–	12.0	–						
Proportional calc.	0.273	–	12.0	–						
Rounded up step	0.3	–	12.0	–						
Abstract trials										
1	2.8	3.75	78	90	5.75	6.10	5.49	5.90	5.81	5.041
2	3.1	3.75	90	90	6.10	4.50	6.60	6.40	5.90	–
3	3.4	3.75	102	90	5.62	6.30	6.25	5.62	5.95	6.073
4	3.7	3.75	114	90	6.40	7.00	6.00	5.50	6.22	6.589
5	4.0	3.75	126	90	5.60	6.90	6.35	5.75	6.15	7.105
6	4.3	3.75	138	90	6.10	6.60	6.75	6.75	6.55	7.621
7	4.6	3.75	150	90	6.65	6.35	5.90	6.75	6.41	8.130
8	4.9	3.75	162	90	5.65	5.45	5.75	6.00	5.71	–

2.29

Name	x_1	x_2	x_3	Response	
Basic level	1.500	1.100	3.900		
Variation interval	0.300	0.200	0.900		
Regression coefficients	0.00987	-0.00275	-0.00225		
Product $b_i \times \Delta x_i$	0.002961	-0.00055	-0.002025		
Step in change x_1 for 0.36	+0.36	-0.067	-0.246		
Rounded up step	+0.36	-0.07	-0.25		
Trials	X_1	X_2	X_3	y_u	$\hat{y}$
1	1.86	1.03	3.65	0.259	0.275
2	2.22	0.96	3.40	0.249	0.261
3	2.58	0.89	3.15	0.247	0.248
4	2.94	0.82	2.90	0.245	0.235
5	3.30	0.75	2.65	0.243	0.221
6	3.66	0.68	2.40	0.242	0.208
7	4.02	0.61	2.15	0.244	0.194
8	4.38	0.54	1.90	0.246	0.181

Basic level			3.66	0.68	2.40							
Lower level			3.16	0.38	1.50							
Upper level			4.16	0.98	3.30							
Variation interval			0.50	0.30	0.90							
No.	X_0	X_1	X_2	X_3	X_1^2	X_2^2	X_3^2	X_1X_2	X_1X_3	X_2X_3	y_u	$\widehat{y}$
1	+	−	−	−	+	+	+	+	+	+	0.258	0.257
2	+	+	−	−	+	+	+	−	−	+	0.280	0.276
3	+	−	+	−	+	+	+	−	+	−	0.271	0.275
4	+	+	+	−	+	+	+	+	−	−	0.272	0.276
5	+	−	−	+	+	+	+	+	−	−	0.247	0.244
6	+	+	−	+	+	+	+	−	+	−	0.255	0.252
7	+	−	+	+	+	+	+	−	−	+	0.253	0.257
8	+	+	+	+	+	+	+	+	+	+	0.246	0.248
9	+	-1.682	0	0	2.828	0	0	0	0	0	0.267	0.265
10	+	1.682	0	0	2.828	0	0	0	0	0	0.271	0.279
11	+	0	-1.682	0	0	2.828	0	0	0	0	0.251	0.246
12	+	0	1.682	0	0	2.828	0	0	0	0	0.253	0.257
13	+	0	0	-1.682	0	0	2.828	0	0	0	0.278	0.276
14	+	0	0	1.682	0	0	2.828	0	0	0	0.241	0.241
15	+	0	0	0	0	0	0	0	0	0	0.242	0.242
16	+	0	0	0	0	0	0	0	0	0	0.232	0.242
17	+	0	0	0	0	0	0	0	0	0	0.235	0.242
18	+	0	0	0	0	0	0	0	0	0	0.243	0.242
19	+	0	0	0	0	0	0	0	0	0	0.251	0.242
20	+	0	0	0	0	0	0	0	0	0	0.249	0.242

DropBooks

Regression coefficients have the values:

$b_0 = 0.242;$ $b_1 = 0.0023;$ $b_2 = 0.00347;$ $b_3 = -0.01026;$
$b_{11} = 0.00944;$ $b_{22} = 0.0033;$ $b_{33} = 0.00587;$ $b_{12} = -0.00429;$
$b_{13} = -0.00261;$ $b_{23} = -0.001.$

2.30

$$y - 463.53 = 25.21Z_1^2 + 76.17Z_2^2$$
$$Z_1 = 0.995(X_1 - 1.40) + 0.099(X_2 - 0.55)$$
$$Z_2 = 0.099(X_1 - 1.40) - 0.995(X_2 - 0.55)$$

2.31

$$y - 52.12 = 0.35Z_1^2 - 1.85Z_2^2$$

2.32

$$y_1 - 22.5 = 12.5Z_1^2 + 6.9Z_2^2$$
$$y_2 - 64.6 = 1.8Z_1^2 - 11.1Z_2^2$$

A.2
Tables of Statistical Functions

Table A Random numbers

83 28	78 05	18 98	49 22	54 11	92 37	45 11	63 60	19 05	91 26
84 73	82 58	01 90	55 37	85 68	98 15	99 52	99 84	51 91	73 81
00 79	20 99	42 57	55 67	93 39	99 25	65 10	91 54	84 65	16 23
94 48	02 99	71 08	50 84	66 10	10 34	92 30	89 28	30 74	24 24
54 37	52 43	87 22	21 34	20 15	07 67	64 98	36 01	33 34	04 42
47 68	59 90	98 90	27 71	89 89	98 20	24 19	85 02	34 38	26 71
76 16	58 55	51 85	44 00	28 28	38 91	70 70	16 81	13 49	46 54
37 64	90 35	64 45	47 72	82 03	01 65	05 97	13 90	90 57	51 97
92 78	39 12	48 01	83 46	39 29	98 71	39 56	97 66	97 70	05 77
24 50	29 02	71 28	53 99	75 07	13 18	76 97	72 54	85 79	71 60
01 72	71 23	86 40	70 05	35 36	15 64	11 01	11 18	90 14	95 05
43 28	52 77	22 80	49 89	79 65	91 17	80 94	34 02	17 61	00 42
29 09	19 54	67 67	88 54	62 09	07 97	35 19	31 25	06 92	25 02
27 95	74 89	62 45	75 39	06 89	58 96	64 65	81 84	85 20	01 47
52 43	54 97	75 80	00 38	20 38	57 46	57 33	87 19	66 06	40 32
78 11	60 42	09 83	28 40	93 57	61 22	27 27	47 80	44 34	47 27
03 74	36 27	13 19	14 76	35 73	66 29	95 65	12 87	61 91	34 30
82 25	35 57	16 29	21 27	51 23	06 52	40 00	28 11	47 23	63 01
09 91	87 20	33 76	61 55	79 21	74 36	21 36	05 47	28 42	92 51
19 82	00 40	15 52	45 35	13 48	74 10	97 36	22 85	44 57	91 72
69 41	17 07	11 54	36 81	57 38	55 39	85 74	48 05	06 43	10 63
48 80	36 26	28 95	03 79	54 31	41 55	48 84	78 63	09 05	69 07
80 02	51 78	94 07	88 62	85 82	80 37	56 15	59 30	46 42	84 02
19 51	95 22	72 72	95 51	57 73	04 68	00 95	04 30	66 52	60 74
50 36	31 76	75 39	04 95	69 47	95 23	01 70	95 04	04 18	68 14
60 03	34 57	41 76	35 06	75 60	21 58	86 36	02 33	00 59	63 13
59 40	60 83	61 73	45 18	08 23	54 86	64 57	76 70	00 89	43 24
29 51	12 43	14 24	35 78	76 22	82 50	68 02	13 19	07 00	19 07
57 07	34 86	57 96	99 57	44 54	90 87	33 76	71 71	23 28	88 37
81 73	29 08	96 62	34 26	52 32	23 74	17 49	45 62	17 88	50 50
40 20	21 54	17 65	99 31	09 72	67 87	16 34	00 76	26 23	42 40
81 26	86 30	79 17	93 45	74 50	50 24	65 52	06 59	04 60	73 63
13 65	31 57	36 88	98 35	04 96	41 37	45 87	57 57	21 15	34 59
23 41	47 66	24 73	31 96	72 07	09 43	88 63	33 80	54 79	84 18
79 62	53 27	85 43	5I 69	83 81	90 85	84 72	18 48	41 20	81 59
l3 40	75 73	19 92	12 01	91 95	23 99	99 30	30 58	46 22	64 41
54 87	97 55	83 91	42 61	41 02	40 18	39 20	56 19	56 35	04 32
09 29	30 63	75 86	85 29	15 34	68 92	34 06	81 60	32 16	05 37
61 99	27 99	73 18	94 29	25 74	22 20	70 46	30 38	26 91	59 16
31 84	93 27	40 23	25 86	68 30	10 11	91 59	61 07	41 97	10 39
35 86	11 25	98 38	27 14	79 68	77 60	63 34	23 80	75 43	48 79
40 42	68 85	23 40	27 56	54 56	75 65	70 49	24 08	10 44	75 59
25 14	94 00	99 80	81 44	49 08	98 93	71 74	11 14	54 69	71 69
56 18	75 63	56 68	25 36	75 98	00 18	19 15	24 28	56 80	75 97
79 61	54 67	58 38	93 69	45 95	61 19	17 35	89 90	98 70	26 20
92 91	85 49	33 32	46 67	28 20	40 99	88 73	56 33	29 13	41 89
01 79	85 45	45 36	05 67	56 17	59 77	59 34	35 01	15 21	00 35
55 84	71 36	40 39	47 25	25 73	69 14	55 73	35 86	61 17	98 69
38 36	66 66	19 40	90 83	06 31	24 67	91 74	54 14	87 24	61 80
01 69	50 70	31 02	98 86	42 01	94 98	07 85	28 38	37 30	72 76

Table A (continued)

87 08	83 09	40 14	39 15	99 24	21 85	00 45	54 19	36 18	03 88
88 33	78 20	40 40	24 73	77 70	00 31	84 59	26 06	50 30	95 96
22 50	09 11	00 37	36 51	55 95	83 97	13 75	46 22	77 50	11 72
48 70	56 57	16 24	21 74	91 53	18 05	59 61	74 97	31 82	77 68
93 45	40 93	12 80	88 63	26 93	85 05	19 87	84 37	59 76	16 65
50 76	72 02	39 19	40 69	57 23	09 33	20 70	86 45	13 94	98 39
91 64	01 34	67 13	11 00	32 09	39 76	21 64	29 85	65 14	51 74
33 20	63 71	95 94	13 77	12 44	12 94	91 04	41 83	79 72	44 08
90 59	65 46	78 82	16 45	97 85	57 75	79 96	79 08	16 83	43 99
05 10	93 57	80 32	86 65	26 90	27 54	34 94	46 33	65 35	56 84
92 85	63 26	69 69	81 54	70 56	17 62	43 17	86 78	99 62	34 15
08 50	36 55	82 11	26 54	76 88	85 67	82 21	65 00	83 89	06 09
59 36	77 09	83 87	81 77	93 77	48 44	88 30	37 21	74 02	93 10
05 85	86 43	25 50	76 70	36 32	26 68	54 92	84 90	02 38	77 40
13 46	99 31	30 29	71 70	91 10	99 84	55 31	95 20	90 28	49 78
56 27	09 33	66 79	32 29	50 54	76 94	27 01	45 87	29 66	23 15
54 15	62 11	22 33	39 39	58 30	73 43	59 32	26 43	76 12	99 10
83 01	86 58	89 77	68 87	29 71	49 50	46 53	56 53	41 53	52 20
00 28	17 33	81 42	24 33	55 75	42 70	73 65	16 96	47 17	42 69
52 29	68 59	32 69	40 30	89 12	11 07	18 53	27 13	46 54	85 40
64 43	09 80	68 29	86 65	60 27	87 70	77 45	31 69	12 31	21 79
80 68	13 48	80 84	25 33	70 89	76 61	03 41	57 89	87 07	56 12
28 72	57 80	54 05	80 92	82 65	25 01	74 58	89 39	25 05	57 66
33 48	49 96	00 17	88 90	63 67	02 64	71 12	21 02	29 86	88 54
04 41	27 70	10 49	13 76	99 38	64 14	90 60	69 75	10 97	16 60
21 31	95 96	89 48	65 14	12 02	94 50	35 64	58 43	92 07	74 08
52 08	13 32	36 45	39 54	82 26	46 60	04 19	34 61	36 12	46 15
90 57	88 69	61 05	22 76	90 79	01 74	22 08	26 13	95 13	75 53
76 50	49 80	25 61	81 96	19 92	33 14	60 41	27 06	05 98	51 49
06 84	76 10	54 41	54 56	15 96	49 19	65 51	93 32	54 54	95 67
47 92	60 37	45 39	67 64	70 05	06 54	84 10	88 68	33 60	77 81
71 87	94 13	64 75	18 17	76 80	95 10	33 33	35 31	30 47	53 74
38 30	36 79	74 83	61 91	56 22	83 73	15 54	63 39	50 33	88 83
09 80	50 48	23 26	05 85	68 97	06 78	00 17	76 05	95 31	03 37
82 52	08 00	33 76	29 14	18 59	98 12	89 34	50 70	13 07	60 38
14 18	02 28	72 80	85 72	09 59	05 26	05 26	90 65	47 12	85 65
62 60	63 74	20 31	60 66	90 87	09 41	59 73	60 00	21 96	38 40
15 02	56 81	29 34	90 99	07 57	80 24	92 41	88 41	01 88	05 62
23 32	03 76	20 25	96 68	01 99	79 82	58 06	89 54	74 06	01 39
96 66	81 45	01 09	18 35	41 97	70 37	94 95	48 64	01 75	04 39
12 41	98 35	82 38	49 91	71 57	83 06	55 84	38 04	70 18	75 19
70 78	63 95	94 82	54 88	47 69	63 32	79 75	3l 56	38 92	54 43
30 43	70 43	70 32	73 47	49 64	23 54	59 17	80 48	61 66	45 66
36 58	96 32	60 46	60 87	52 75	53 13	39 19	41 52	24 14	88 93
17 35	36 91	90 59	48 78	99 31	64 40	84 05	79 00	53 03	64 02
73 30	27 77	44 50	07 79	27 66	42 39	97 64	84 36	18 13	59 61
92 15	47 21	82 54	76 05	54 10	40 93	71 96	66 52	83 98	17 85
05 02	28 36	50 64	47 21	36 25	80 01	43 41	36 58	97 15	29 95
5l 22	04 71	06 37	31 45	69 62	30 84	20 28	14 41	70 05	56 88
23 28	85 05	96 40	37 56	52 60	65 75	21 47	84 15	99 92	02 41

DropBooks

Table B Standardized normal distribution

x	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
	0,	0,	0,	0,	0,	0,	0,	0,	0,	0,
0.0	5000	5040	5080	5120	5160	5199	5239	5279	5319	5359
0.1	5398	5438	5478	5517	5557	5596	5636	5675	5714	5753
0.2	5793	5832	5871	5910	5948	5987	6026	6064	6103	6141
0.3	6179	6217	6255	6293	6331	6368	6406	6443	6480	6517
0.4	6554	6591	6628	6664	6700	6736	6772	6808	6844	6879
0.5	6915	6950	6985	7019	7054	7088	7123	7157	7190	7224
0.6	7257	7291	7324	7357	7389	7422	7454	7486	7517	7549
0.7	7580	7611	7642	7673	7704	7734	7764	7794	7823	7852
0.8	7881	7910	7939	7967	7995	8023	8051	8078	8106	8133
0.9	8159	8186	8212	8238	8264	8289	8315	8340	8365	8389
1.0	8413	8438	8461	8485	8508	8531	8554	8577	8599	8621
1.1	8643	8665	8686	8708	8729	8749	8770	8790	8810	8830
1.2	8849	8869	8888	8907	8925	8944	8962	8980	8997	9015
1.3	9032	9049	9066	9082	9099	9115	9131	9147	9162	9177
1.4	9192	9207	9222	9236	9251	9265	9279	9292	9306	9319
1.5	9332	9345	9357	9370	9382	9394	9406	9418	9429	9441
1.6	9452	9463	9474	9484	9495	9505	9515	9525	9535	9545
1.7	9554	9564	9573	9582	9591	9599	9608	9616	9616	9633
1.8	9641	9649	9656	9664	9671	9678	9686	9693	9699	9706
1.9	9713	9719	9726	9732	9738	9744	9750	9756	9761	9767
2.0	9772	9778	9783	9788	9793	9798	9803	9808	9812	9817
2.1	9821	9826	9830	9834	9838	9842	9846	9850	9854	9857
2.2	9861	9864	9868	9871	9875	9878	9881	9884	9887	9890
2.3	9893	9896	9898	9901	9904	9906	9909	9911	9913	9916
2.4	9918	9920	9922	9925	9927	9929	9931	9932	9934	9936
2.5	9938	9940	9941	9943	9945	9946	9948	9949	9951	9952
2.6	9953	9955	9956	9957	9959	9960	9961	9962	9963	9964
2.7	9965	9966	9967	9968	9969	9970	9971	9972	9973	9974
2.8	9974	9975	9976	9977	9977	9978	9979	9979	9980	9981
2.9	9981	9982	9982	9983	9984	9984	9985	9985	9986	9986
3.0	9987	9987	9987	9988	9988	9989	9989	9989	9990	9990
3.1	9990	9991	9991	9991	9992	9992	9992	9992	9993	9993
3.2	9993	9993	9994	9994	9994	9994	9994	9995	3995	9995
3.3	9995	9995	9995	9996	9996	9996	9996	9996	9996	9997
3.4	9997	9997	9997	9997	9997	9997	9997	9997	9997	9998

Table C Student's t-distribution

f	α/2				
	0.1	**0.05**	**0.025**	**0.01**	**0.005**
1	3.08	6.31	12.70	31.80	63.70
2	1.89	2.92	4.30	6.96	9.92
3	1.64	2.35	3.18	4.54	5.84
4	1.53	2.13	2.78	3.75	4.60
5	1.48	2.01	2.57	3.36	4.03
6	1.44	1.94	2.45	3.14	3.71
7	1.42	1.89	2.36	3.00	3.50
8	l,40	1.86	2.31	2.90	3.36
9	1.38	1.83	2.26	2.82	3.25
10	1.37	1.8l	2.23	2.76	3.17
11	1.36	1.80	2.20	2.72	3.11
I2	1.36	1.78	2.18	2.68	3.05
13	1.35	1.77	2.16	2.65	3.01
14	1.34	1.76	2.14	2.62	2.98
15	1.34	1.75	2.13	2.60	2.95
16	1.34	1.75	2.12	2.58	2.92
17	1.33	1.74	2.11	2.57	2.90
18	1.33	1.73	2.10	2.55	2.88
19	1.33	1.73	2.09	2.54	2.86
20	1.32	1.72	2.09	2.53	2.85
21	1.32	1.72	2.08	2.52	2.83
22	1.32	1.72	2.07	2.51	2.82
23	1.32	1.71	2.07	2.50	2.81
24	1.32	1.71	2.06	2.49	2.80
25	1.32	1.7l	2.06	2.48	2.79
26	1.32	1.71	2.06	2.48	2.78
27	1.31	1.70	2.05	2.47	2.77
28	1.31	1.70	2.05	2.47	2.76
29	1.31	1.70	2.05	2.46	2.76
30	1.31	1.70	2.04	2.46	2.75
40	1.30	1.68	2.02	2.42	2.70
60	1.30	1.67	2.00	2.39	2.66
120	1.29	1.66	1.98	2.36	2.62
∞	1.28	1.64	1.96	2.33	2.58

DropBooks

Table D Chi-square distribution

	P														
f	**0.995**	**0.99**	**0.975**	**0.95**	**0.9**	**0.75**	**0.5**	**0.25**	**0.1**	**0.05**	**0.025**	**0.01**	**0.005**	**0.001**	**f**
1	–	–	–	–	0.016	0.102	0.455	1.32	2.71	3.84	5.02	6.63	7.88	10.8	1
2	0.010	0.020	0.051	0.103	0.211	0.575	1.39	2.77	4.61	5.99	7.38	9.21	10.6	13.8	2
3	0.072	0.115	0.216	0.352	0.584	1.21	2.37	4.11	6.25	7.81	9.35	11.3	12.8	16.3	3
4	0.207	0.297	0.484	0.711	1.06	1.92	3.36	5.39	7.78	9.49	11.1	13.3	14.9	18.5	4
5	0.412	0.554	0.831	1.15	1.61	2.67	4.35	6.63	9.24	11.1	12.8	15.1	16.7	20.5	5
6	0.676	0.872	1.24	1.64	2.20	3.45	5.35	7.84	10.6	12.6	14.4	16.8	18.5	22.5	6
7	0.989	1.24	1.69	2.17	2.83	4.25	6.35	9.04	12.0	14.1	16.0	18.5	20.3	24.3	7
8	1.34	1.65	2.18	2.73	3.49	5.07	7.34	10.2	13.4	15.5	17.5	20.1	22.0	26.1	8
9	1.73	2.09	2.70	3.33	4.17	5.90	8.34	11.4	14.7	16.9	19.0	21.7	23.6	27.9	9
10	2.16	2.56	3.25	3.94	4.87	6.74	9.34	12.5	16.0	18.3	20.5	23.2	25.2	29.6	10
11	2.60	3.05	3.82	4.57	5.58	7.58	10.3	13.7	17.3	19.7	21.9	24.7	26.8	31.3	11
12	3.07	3.57	4.40	5.23	6.30	8.44	11.3	14.8	18.5	21.0	23.3	26.2	28.3	32.9	12
13	3.57	4.11	5.01	5.89	7.04	9.30	12.3	16.0	19.8	22.4	24.7	27.7	29.8	34.5	13
14	4.07	4.66	5.63	6.57	7.79	10.2	13.3	17.1	21.1	23.7	26.1	29.1	31.3	36.1	14
15	4.60	5.23	6.26	7.26	8.55	11.0	14.3	18.2	22.3	25.0	27.5	30.6	32.8	37.7	15
16	5.14	5.81	6.91	7.96	9.31	11.9	15.3	19.4	23.5	26.3	28.8	32.0	34.3	39.3	16
17	5.70	6.41	7.56	8.67	10.1	12.8	16.3	20.5	24.8	27.6	30.2	33.4	35.7	40.8	17
18	6.26	7.01	8.23	9.39	10.9	13.7	17.3	21.6	26.0	28.9	31.5	34.8	37.2	42.3	18
19	6.84	7.63	8.91	10.1	11.7	14.6	18.3	22.7	27.2	30.1	32.9	36.2	38.6	43.8	19
20	7.43	8.26	9.59	10.9	12.4	15.5	19.3	23.8	28.4	31.4	34.2	37.6	40.0	45.3	20
21	8.03	8.90	10.3	11.6	13.2	16.3	20.3	24.9	29.6	32.7	35.5	38.9	41.4	46.8	21
22	8.64	9.54	11.0	12.3	14.0	17.2	21.3	26.0	30.8	33.9	36.8	40.3	42.8	48.3	22
23	9.26	10.2	11.7	13.1	14.8	18.1	22.3	27.1	32.0	35.2	38.1	41.6	44.2	49.7	23
24	9.89	10.9	12.4	13.8	15.7	19.0	23.3	28.2	33.2	36.4	39.4	43.0	45.6	51.2	24
25	10.5	11.5	13.1	14.6	16.5	19.9	24.3	29.3	34.4	37.7	40.6	44.3	46.9	52.6	25
26	11.2	12.2	13.8	15.4	17.3	20.8	25.3	30.4	35.6	38.9	41.9	45.6	48.3	54.1	26
27	11.8	12.9	14.6	16.2	18.1	21.7	26.3	31.5	36.7	40.1	43.2	47.0	49.6	55.5	27
28	12.5	13.6	15.3	16.9	18.9	22.7	27.3	32.6	37.9	41.3	44.5	48.3	51.0	56.9	28
29	13.1	14.3	16.0	17.7	19.8	23.6	28.3	33.7	39.1	42.6	45.7	49.6	52.3	58.3	29
30	13.8	15.0	16.8	18.5	20.6	24.5	29.3	34.8	40.3	43.8	47.0	50.9	53.7	59.7	30

Table E F-distribution

α	f_2	f_1 (for greater variance)																		f_2	α
		1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40	60	120	∞	
0.1		39.9	49.5	53.6	55.8	57.2	58.2	58.9	59.4	59.9	60.2	60.7	61.2	61.7	62.0	62.3	62.5	62.8	63.1	63.3		0.1
0.05	1	161	199	216	225	230	234	237	239	241	242	244	246	248	249	250	251	252	253	254	1	0.05
0.01		4052	4999	5403	5625	5764	5859	5928	5982	6022	6056	6106	6157	6209	6235	6261	6287	6313	6339	6366		0.01
0.1		8.53	9.00	9.16	9.24	9.29	9.33	9.35	9.37	9.38	9.39	9.41	9.42	9.44	9.45	9.46	9.47	9.47	9.48	9.49		0.1
0.05	2	18.5	19.0	19.2	19.2	19.3	19.3	19.4	19.4	19.4	19.4	19.4	19.4	19.4	19.5	19.5	19.5	19.5	19.5	19.5	2	0.05
0.01		98.5	99.0	99.2	99.2	99.3	99.3	99.4	99.4	99.4	99.4	99.4	99.4	99.4	99.5	99.5	99.5	99.5	99.5	99.5		0.01
0.1		5.54	5.46	5.39	5.34	5.31	5.28	5.27	5.25	5.24	5.23	5.22	5.20	5.18	5.18	5.17	5.16	5.15	5.14	5.13		0.1
0.05	3	10.1	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81	8.79	8.74	8.70	8.66	8.64	8.62	8.59	8.57	8.55	8.53	3	0.05
0.01		34.1	30.8	29.5	28.7	28.2	27.9	27.7	27.5	27.3	27.2	27.1	26.9	26.7	26.6	26.5	26.4	26.3	26.2	26.1		0.01
0.1		4.54	4.32	4.19	4.11	4.05	4.01	3.98	3.95	3.94	3.92	3.90	3.87	3.84	3.83	3.82	3.80	3.79	3.78	3.76		0.1
0.05	4	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.91	5.86	5.80	5.77	5.75	5.72	5.69	5.66	5.63	4	0.05
0.01		21.2	18.0	16.7	16.0	15.5	15.2	15.0	14.8	14.7	14.5	14.4	14.2	14.0	13.9	13.8	13.7	13.7	13.6	13.5		0.01
0.1		4.06	3.78	3.62	3.52	3.45	3.40	3.37	3.34	3.32	3.30	3.27	3.24	3.21	3.19	3.17	3.16	3.14	3.12	3.10		0.1
0.05	5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77	4.74	4.68	4.62	4.56	4.53	4.50	4.46	4.43	4.40	4.36	5	0.05
0.01		16.3	13.3	12.1	11.4	11.0	10.7	10.5	10.3	10.2	10.1	9.89	9.72	9.55	9.47	9.38	9.29	9.20	9.11	9.02		0.01
0.1		3.78	3.46	3.29	3.18	3.11	3.05	3.01	2.98	2.96	2.94	2.90	2.87	2.84	2.82	2.80	2.78	2.76	2.74	2.72		0.1
0.05	6	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	4.00	3.94	3.87	3.84	3.81	3.77	3.74	3.70	3.67	6	0.05
0.01		13.7	10.9	9.78	9.15	8.75	8.47	8.26	8.10	7.98	7.87	7.72	7.56	7.40	7.31	7.23	7.14	7.06	6.97	6.88		0.01
0.1		3.59	3.26	3.07	2.96	2.88	2.83	2.78	2.75	2.72	2.70	2.67	2.63	2.59	2.58	2.56	2.54	2.51	2.49	2.47		0.1
0.05	7	5.59	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.64	3.57	3.51	3.44	3.41	3.38	3.34	3.30	3.27	3.23	7	0.05
0.01		12.2	9.55	8.45	7.85	7.46	7.19	6.99	6.84	6.72	6.62	6.47	6.31	6.16	6.07	5.99	5.91	5.82	5.74	5.65		0.01
0.1		3.46	3.11	2.92	2.81	2.73	2.67	2.62	2.59	2.56	2.54	2.50	2.46	2.42	2.40	2.38	2.36	2.34	2.32	2.29		0.1
0.05	8	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.35	3.28	3.22	3.15	3.12	3.08	3.04	3.01	2.97	2.93	8	0.05
0.01		11.3	8.65	7.59	7.01	6.63	6.37	6.18	6.03	5.91	5.81	5.67	5.52	5.36	5.28	5.20	5.12	5.03	4.95	4.86		0.01
0.1		3.36	3.01	2.81	2.69	2.61	2.55	2.51	2.47	2.44	2.42	2.38	2.34	2.30	2.28	2.25	2.23	2.21	2.18	2.16		0.1
0.05	9	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.14	3.07	3.01	2.94	2.90	2.86	2.83	2.79	2.75	2.71	9	0.05
0.01		10.6	8.02	6.99	6.42	6.06	5.80	5.61	5.47	5.35	5.26	5.11	4.96	4.81	4.73	4.65	4.57	4.48	4.40	4.31		0.01

Springer Nature

Table E (continued)

α	f_2	f_1(for greater variance)																			f_2	α
		1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40	60	120	∞		
0.1		3.28	2.92	2.73	2.61	2.52	2.46	2.41	2.38	2.35	2.32	2.28	2.24	2.20	2.18	2.16	2.13	2.11	2.08	2.06		0.1
0.05	10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.98	2.91	2.84	2.77	2.74	2.70	2.66	2.62	2.58	2.54	10	0.05
0.01		10.0	7.56	6.55	5.99	5.64	5.39	5.20	5.06	4.94	4.85	4.71	4.56	4.41	4.33	4.25	4.17	4.08	4.00	3.91		0.01
0.1		3.23	2.86	2.66	2.54	2.45	2.39	2.34	2.30	2.27	2.25	2.21	2.17	2.12	2.10	2.08	2.05	2.03	2.00	1.97		0.1
0.05	11	4.84	3.98	3.59	3.36	3.20	3.09	3.01	2.95	2.90	2.85	2.79	2.72	2.65	2.61	2.57	2.53	2.49	2.45	2.40	11	0.05
0.01		9.65	7.21	6.22	5.67	5.32	5.07	4.89	4.74	4.63	4.54	4.40	4.25	4.10	4.02	3.94	3.86	3.78	3.69	3.60		0.01
0.1		3.18	2.81	2.61	2.48	2.39	2.33	2.28	2.24	2.21	2.19	2.15	2.10	2.06	2.04	2.01	1.99	1.96	1.93	1.90		0.1
0.05	12	4.75	3.89	3.49	3.26	3.11	3.00	2.91	2.85	2.80	2.75	2.69	2.62	2.54	2.51	2.47	2.43	2.38	2.34	2.30	12	0.05
0.01		9.33	6.93	5.95	5.41	5.06	4.82	4.64	4.50	4.39	4.30	4.16	4.01	3.86	3.78	3.70	3.62	3.54	3.45	3.36		0.01
0.1		3.14	2.76	2.56	2.43	2.35	2.28	2.23	2.20	2.16	2.14	2.10	2.05	2.01	1.98	1.96	1.93	1.90	1.88	1.85		0.1
0.05	13	4.67	3.81	3.41	3.18	3.03	2.92	2.83	2.77	2.71	2.67	2.60	2.53	2.46	2.42	2.38	2.34	2.30	2.25	2.21	13	0.05
0.01		9.07	6.70	5.74	5.21	4.86	4.62	4.44	4.30	4.19	4.10	3.96	3.82	3.66	3.59	3.51	3.43	3.34	3.25	3.17		0.01
0.1		3.10	2.73	2.52	2.39	2.31	2.24	2.19	2.15	2.12	2.10	2.05	2.01	1.96	1.94	1.91	1.89	1.86	1.83	1.80		0.1
0.05	14	4.60	3.74	3.34	3.11	2.96	2.85	2.76	2.70	2.65	2.60	2.53	2.46	2.39	2.35	2.31	2.27	2.22	2.18	2.13	14	0.05
0.01		8.86	6.51	5.56	5.04	4.69	4.46	4.28	4.14	4.03	3.94	3.80	3.66	3.51	3.43	3.35	3.27	3.18	3.09	3.00		0.01
0.1		3.07	2.70	2.49	2.36	2.27	2.21	2.16	2.12	2.09	2.06	2.02	1.97	1.92	1.90	1.87	1.85	1.82	1.79	1.76		0.1
0.05	15	4.54	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59	2.54	2.48	2.40	2.33	2.29	2.25	2.20	2.16	2.11	2.07	15	0.05
0.01		8.68	6.36	5.42	4.89	4.56	4.32	4.14	4.00	3.89	3.80	3.67	3.52	3.37	3.29	3.21	3.13	3.05	2.96	2.87		0.01
0.1		3.05	2.67	2.46	2.33	2.24	2.18	2.13	2.09	2.06	2.03	1.99	1.94	1.89	1.87	1.84	1.81	1.78	1.75	1.72		0.1
0.05	16	4.49	3.63	3.24	3.01	2.85	2.74	2.66	2.59	2.54	2.49	2.42	2.35	2.28	2.24	2.19	2.15	2.11	2.06	2.01	16	0.05
0.01		8.53	6.23	5.29	4.77	4.44	4.20	4.03	3.89	3.78	3.69	3.55	3.41	3.26	3.18	3.10	3.02	2.93	2.84	2.75		0.01
0.1		3.03	2.64	2.44	2.31	2.22	2.15	2.10	2.06	2.03	2.00	1.96	1.91	1.86	1.84	1.81	1.78	1.75	1.72	1.69		0.1
0.05	17	4.45	3.59	3.20	2.96	2.81	2.70	2.61	2.55	2.49	2.45	2.38	2.31	2.23	2.19	2.15	2.10	2.06	2.01	1.96	17	0.05
0.01		8.40	6.11	5.18	4.67	4.34	4.10	3.93	3.79	3.68	3.59	3.46	3.31	3.16	3.08	3.00	2.92	2.83	2.75	2.65		0.01
0.1		3.01	2.62	2.42	2.29	2.20	2.13	2.08	2.04	2.00	1.98	1.93	1.89	1.84	1.81	1.78	1.75	1.72	1.69	1.66		0.1
0.05	18	4.41	3.55	3.16	2.93	2.77	2.66	2.58	2.51	2.46	2.41	2.34	2.27	2.19	2.15	2.11	2.06	2.02	1.97	1.92	18	0.05
0.01		8.29	6.01	5.09	4.58	4.25	4.01	3.84	3.71	3.60	3.51	3.37	3.23	3.08	3.00	2.92	2.84	2.75	2.66	2.57		0.01

Table E (continued)

α	f_2	f_1 (for greater variance)																		f_2	α
		1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40	60	120	∞	
0.1		2.99	2.61	2.40	2.27	2.18	2.11	2.06	2.02	1.98	1.96	1.91	1.86	1.81	1.79	1.76	1.73	1.70	1.67	1.63	0.1
0.05	19	4.38	3.52	3.13	2.90	2.74	2.63	2.54	2.48	2.42	2.38	2.31	2.23	2.16	2.11	2.07	2.03	1.98	1.93	1.88	19	0.05
0.01		8.18	5.93	5.01	4.50	4.17	3.94	3.77	3.63	3.52	3.43	3.30	3.15	3.00	2.92	2.84	2.76	2.67	2.58	2.49		0.01
0.1		2.97	2.59	2.38	2.25	2.16	2.09	2.04	2.00	1.96	1.94	1.89	1.84	1.79	1.77	1.74	1.71	1.68	1.64	1.61		0.1
0.05	20	4.35	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39	2.35	2.28	2.20	2.12	2.08	2.04	1.99	1.95	1.90	1.84	20	0.05
0.01		8.10	5.85	4.94	4.43	4.10	3.87	3.70	3.56	3.46	3.37	3.23	3.09	2.94	2.86	2.78	2.69	2.61	2.52	2.42		0.01
0.1		2.96	2.57	2.36	2.23	2.14	2.08	2.02	1.98	1.95	1.92	1.87	1.83	1.78	1.75	1.72	1.69	1.66	1.62	1.59		0.1
0.05	21	4.32	3.47	3.07	2.84	2.68	2.57	2.49	2.42	2.37	2.32	2.25	2.18	2.10	2.05	2.01	1.96	1.92	1.87	1.81	21	0.05
0.01		8.02	5.78	4.87	4.37	4.04	3.81	3.64	3.51	3.40	3.31	3.17	3.03	2.88	2.80	2.72	2.64	2.55	2.46	2.36		0.01
0.1		2.95	2.56	2.35	2.22	2.13	2.06	2.01	1.97	1.93	1.90	1.86	1.81	1.76	1.73	1.70	1.67	1.64	1.60	1.57		0.1
0.05	22	4.30	3.44	3.05	2.82	2.66	2.55	2.46	2.40	2.34	2.30	2.23	2.15	2.07	2.03	1.98	1.94	1.89	1.84	1.78	22	0.05
0.01		7.95	5.72	4.82	4.31	3.99	3.76	3.59	3.45	3.35	3.26	3.12	2.98	2.83	2.75	2.67	2.58	2.50	2.40	2.31		0.01
0.1		2.94	2.55	2.34	2.21	2.11	2.05	1.99	1.95	1.92	1.89	1.85	1.80	1.74	1.72	1.69	1.66	1.62	1.59	1.55		0.1
0.05	23	4.28	3.42	3.03	2.80	2.64	2.53	2.44	2.37	2.32	2.27	2.20	2.13	2.05	2.00	1.96	1.91	1.86	1.81	1.76	23	0.05
0.01		7.88	5.66	4.76	4.26	3.94	3.71	3.54	3.41	3.30	3.21	3.07	2.93	2.78	2.70	2.62	2.54	2.45	2.35	2.26		0.01
0.1		2.93	2.54	2.33	2.19	2.10	2.04	1.98	1.94	1.91	1.88	1.83	1.78	1.73	1.70	1.67	1.64	1.61	1.57	1.53		0.1
0.05	24	4.26	3.40	3.01	2.78	2.62	2.51	2.42	2.36	2.30	2.25	2.18	2.11	2.03	1.98	1.94	1.89	1.84	1.79	1.73	24	0.05
0.01		7.82	5.61	4.72	4.22	3.90	3.67	3.50	3.36	3.26	3.17	3.03	2.89	2.74	2.66	2.58	2.49	2.40	2.31	2.21		0.01
0.1		2.92	2.53	2.32	2.18	2.09	2.02	1.97	1.93	1.89	1.87	1.82	1.77	1.72	1.69	1.66	1.63	1.59	1.56	1.52		0.1
0.05	25	4.24	3.39	2.99	2.76	2.60	2.49	2.40	2.34	2.28	2.24	2.16	2.09	2.01	1.96	1.92	1.87	1.82	1.77	1.71	25	0.05
0.01		7.77	5.57	4.68	4.18	3.86	3.63	3.46	3.32	3.22	3.13	2.99	2.85	2.70	2.62	2.54	2.45	2.36	2.27	2.17		0.01
0.1		2.91	2.52	2.31	2.17	2.08	2.01	1.96	1.92	1.88	1.86	1.81	1.76	1.71	1.68	1.65	1.61	1.58	1.54	1.50		0.1
0.05	26	4.23	3.37	2.98	2.74	2.59	2.47	2.39	2.32	2.27	2.22	2.15	2.07	1.99	1.95	1.90	1.85	1.80	1.75	1.69	26	0.05
0.01		7.72	5.53	4.64	4.14	3.82	3.59	3.42	3.29	3.18	3.09	2.96	2.82	2.66	2.58	2.50	2.42	2.33	2.23	2.13		0.01

Springer Books

Table E (continued)

α	f_2	f_1(for greater variance)																			f_2	α
		1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40	60	120	∞		
0.1		2.90	2.51	2.30	2.17	2.07	2.00	1.95	1.91	1.87	1.85	1.80	1.75	1.70	1.67	1.64	1.60	1.57	1.53	1.49		0.1
0.05	27	4.21	3.35	2.96	2.73	2.57	2.46	2.37	2.31	2.25	2.20	2.13	2.06	1.97	1.93	1.88	1.84	1.79	1.73	1.67	27	0.05
0.01		7.68	5.49	4.60	4.11	3.78	3.56	3.39	3.26	3.15	3.06	2.93	2.78	2.63	2.55	2.47	2.38	2.29	2.20	2.10		0.01
0.1		2.89	2.50	2.29	2.16	2.06	2.00	1.94	1.90	1.87	1.84	1.79	1.74	1.69	1.66	1.63	1.59	1.56	1.52	1.48		0.1
0.05	28	4.20	3.34	2.95	2.71	2.56	2.45	2.36	2.29	2.24	2.19	2.12	2.04	1.96	1.91	1.87	1.82	1.77	1.71	1.65	28	0.05
0.01		7.64	5.45	4.57	4.07	3.75	3.53	3.36	3.23	3.12	3.03	2.90	2.75	2.60	2.52	2.44	2.35	2.26	2.17	2.06		0.01
0.1		2.89	2.50	2.28	2.15	2.06	1.99	1.93	1.89	1.86	1.83	1.78	1.73	1.68	1.65	1.62	1.58	1.55	1.51	1.47		0.1
0.05	29	4.18	3.33	2.93	2.70	2.55	2.43	2.35	2.28	2.22	2.18	2.10	2.03	1.94	1.90	1.85	1.81	1.75	1.70	1.64	29	0.05
0.01		7.60	5.42	4.54	4.04	3.73	3.50	3.33	3.20	3.09	3.00	2.87	2.73	2.57	2.49	2.41	2.33	2.23	2.14	2.03		0.01
0.1		2.88	2.49	2.28	2.14	2.05	1.98	1.93	1.88	1.85	1.82	1.77	1.72	1.67	1.64	1.61	1.57	1.54	1.50	1.46		0.1
0.05	30	4.17	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21	2.16	2.09	2.01	1.93	1.89	1.84	1.79	1.74	1.68	1.62	30	0.05
0.01		7.56	5.39	4.51	4.02	3.70	3.47	3.30	3.17	3.07	2.98	2.84	2.70	2.55	2.47	2.39	2.30	2.21	2.11	2.01		0.01
0.1		2.84	2.44	2.23	2.09	2.00	1.93	1.87	1.83	1.79	1.76	1.71	1.66	1.61	1.57	1.54	1.51	1.47	1.42	1.38		0.1
0.05	40	4.08	3.23	2.84	2.61	2.45	2.34	2.25	2.18	2.12	2.08	2.00	1.92	1.84	1.79	1.74	1.69	1.64	1.58	1.51	40	0.05
0.01		7.31	5.18	4.31	3.83	3.51	3.29	3.12	2.99	2.89	2.80	2.66	2.52	2.37	2.29	2.20	2.11	2.02	1.92	1.80		0.01
0.1		2.79	2.39	2.18	2.04	1.95	1.87	1.82	1.77	1.74	1.71	1.66	1.60	1.54	1.51	1.48	1.44	1.40	1.35	1.29		0.1
0.05	60	4.00	3.15	2.76	2.53	2.37	2.25	2.17	2.10	2.04	1.99	1.92	1.84	1.75	1.70	1.65	1.59	1.53	1.47	1.39	60	0.05
0.01		7.08	4.98	4.13	3.65	3.34	3.12	2.95	2.82	2.72	2.63	2.50	2.35	2.20	2.12	2.03	1.94	1.84	1.73	1.60		0.01
0.1		2.75	2.35	2.13	1.99	1.90	1.82	1.77	1.72	1.68	1.65	1.60	1.54	1.48	1.45	1.41	1.37	1.32	1.26	1.19		0.1
0.05	120	3.92	3.07	2.68	2.45	2.29	2.18	2.09	2.02	1.96	1.91	1.83	1.75	1.66	1.61	1.55	1.50	1.43	1.35	1.25	120	0.05
0.01		6.85	4.79	3.95	3.48	3.17	2.96	2.79	2.66	2.56	2.47	2.34	2.19	2.03	1.95	1.86	1.76	1.66	1.53	1.38		0.01
0.1		2.71	2.30	2.08	1.94	1.85	1.77	1.72	1.67	1.63	1.60	1.55	1.49	1.42	1.38	1.34	1.30	1.24	1.17	1.00		0.1
0.05	∞	3.84	3.00	2.60	2.37	2.21	2.10	2.01	1.94	1.88	1.83	1.75	1.67	1.57	1.52	1.46	1.39	1.32	1.22	1.00	∞	0.05
0.01		6.63	4.61	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32	2.18	2.04	1.88	1.79	1.70	1.59	1.47	1.32	1.00		0.01

Table F Number of observations for t-testing of the average

		Significance level																				
		0.01					0.02					0.05					0.1					
One-sided t.		α=0.005					α=0.01					α=0.025					α=0.05					
Two-sided t.		α=0.01					α=0.02					α=0.05					α=0.1					
	β=	0.01	0.05	0.1	0.2	0.5	0.01	0.05	0.1	0.2	0.5	0.01	0.05	0.1	0.2	0.5	0.01	0.05	0.1	0.2	0.5	
	0.05																					0.05
	0.10																					0.10
	0.15																				122	0.15
	0.20										139					99					70	0.20
	0.25					110					90				128	64			139	101	45	0.25
	0.30				134	78				115	63			119	90	45		122	97	71	32	0.30
	0.35			125	99	58			109	85	47		109	88	67	34		90	72	52	24	0.35
	0.40		115	97	77	45		101	85	66	37	117	84	68	51	26	101	70	55	40	19	0.40
D=δ/σ	0.45		92	77	62	37	110	81	68	53	30	93	67	54	41	21	80	55	44	33	15	0.45
	0.50	100	75	63	51	30	90	66	55	43	25	76	54	44	34	18	65	45	36	27	13	0.50
	0.55	83	63	53	42	26	75	55	46	36	21	63	45	37	28	15	54	38	30	22	11	0.55
	0.60	71	53	45	36	22	63	47	39	31	18	53	38	32	24	13	46	32	26	19	9	0.60
	0.65	61	46	39	31	20	55	41	34	27	16	46	33	27	21	12	39	28	22	17	8	0.65
	0.70	53	40	34	28	17	47	35	30	24	14	40	29	24	19	10	34	24	19	15	8	0.70
	0.75	47	36	30	25	16	42	31	27	21	13	35	26	21	16	9	30	21	17	13	7	0.75
	0.80	41	32	27	22	14	37	28	24	19	12	31	22	19	15	9	27	19	15	12	6	0.80
	0.85	37	29	24	20	13	33	25	21	17	11	28	21	17	13	8	24	17	14	11	6	0.85
	0.90	34	26	22	18	12	29	23	19	16	10	25	19	16	12	7	21	15	13	10	5	0.90
	0.95	31	24	20	17	11	27	21	18	14	9	23	17	14	11	7	19	14	11	9	5	0.95
	1.00	28	22	19	16	10	25	19	16	13	9	21	16	13	10	6	18	13	11	8	5	1.00

DropBooks

Table F (continued)

		Significance level																				
		0.01					0.02					0.05					0.1					
One-sided t.		α=0.005					α=0.01					α=0.025					α=0.05					
Two-sided t.		α=0.01					α=0.02					α=0.05					α=0.1					
	β=	0.01	0.05	0.1	0.2	0.5	0.01	0.05	0.1	0.2	0.5	0.01	0.05	0.1	0.2	0.5	0.01	0.05	0.1	0.2	0.5	
	1.1	24	19	16	14	9	21	16	14	12	8	18	13	11	9	6	15	11	9	7		1.1
	1.2	21	16	14	12	8	18	14	12	10	7	15	12	10	8	5	13	10	8	6		1.2
	1.3	18	15	13	11	8	16	13	11	9	6	14	10	9	7		11	8	7	6		1.3
	1.4	16	13	12	10	7	14	11	10	9	6	12	9	8	7		10	8	7	5		1.4
	1.5	15	12	11	9	7	13	10	9	8	6	11	8	7	6		9	7	6			1.5
	1.6	13	11	10	8	6	12	10	9	7	5	10	8	7	6		8	6	6			1.6
	1.7	12	10	9	8	6	11	9	8	7		9	7	6	5		8	6	5			1.7
	1.8	12	10	9	8	6	10	8	7	7		8	7	6			7	6				1.8
D=δ/σ	1.9	11	9	8	7	6	10	8	7	6		8	6	6			7	5				1.9
	2.0	10	8	8	7	5	9	7	7	6		7	6	5			6					2.0
	2.1	10	8	7	7		8	7	6	6		7	6				6					2.1
	2.2	9	8	7	6		8	7	6	5		7	6				6					2.2
	2.3	9	7	7	6		8	6	6			6	5				5					2.3
	2.4	8	7	7	6		7	6	6			6										2.4
	2.5	8	7	6	6		7	6	6			6										2.5
	3.0	7	6	6	5		6	5	5			5										3.0
	3.5	6	5	5			5															3.5
	4.0	6																				

Table G Number of observations for t-testing of difference of averages

	Significance level																				
	0.01					0.02					0.05					0.1					
One-sided t.	α=0.005					α=0.01					α=0.025					α=0.05					
Two-sided t.	α=0.01					α=0.02					α=0.05					α=0.1					
β=	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	
0.05																					0.05
0.10																					0.10
0.15																					0.15
0.20																				137	0.20
0.25															124					88	0.25
0.30										123					87					61	0.30
0.35					110					90					64				102	45	0.35
0.40					85					70				100	50			108	78	35	0.40
D=δ/σ 0.45				118	68				101	55			105	79	39		108	86	62	28	0.45
0.50				96	55			106	82	45		106	86	64	32		88	70	51	23	0.50
0.55			101	79	46		106	88	68	38		87	71	53	27	112	73	58	42	19	0.55
0.60		101	85	67	39		90	74	58	32	104	74	60	45	23	89	61	49	36	16	0.60
0.65		87	73	57	34	104	77	64	49	27	88	63	51	39	20	76	52	42	30	14	0.65
0.70	100	75	63	50	29	90	66	55	43	24	76	55	44	34	17	66	45	36	26	12	0.70
0.75	88	66	55	44	26	79	58	48	38	21	67	48	39	29	15	57	40	32	23	11	0.75
0.80	77	58	49	39	23	70	51	43	33	19	59	42	34	26	14	50	35	28	21	10	0.80
0.85	69	51	43	35	21	62	46	38	30	17	52	37	31	23	12	45	31	25	18	9	0.85
0.90	62	46	39	31	19	55	41	34	27	15	47	34	27	21	11	40	28	22	16	8	0.90
0.95	55	42	35	28	17	50	37	31	24	14	42	30	25	19	10	36	25	20	15	7	0.95
1.00	50	38	32	26	15	45	33	28	22	13	38	27	23	17	9	33	23	18	14	7	1.00

DropBooks

Table G (continued)

	Significance level																					
	0.01					0.02					0.05					0.1					
One-sided t.	α=0.005					α=0.01					α=0.025					α=0.05					
Two-sided t.	α=0.01					α=0.02					α=0.05					α=0.1					
β=	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	**0.01**	**0.05**	**0.1**	**0.2**	**0.5**	
1.1	42	32	27	22	13	38	28	23	19	11	32	23	19	14	18	27	19	15	12	6	1.1
1.2	36	27	23	18	11	32	24	20	16	9	27	20	16	12	7	23	16	13	10	5	1.2
1.3	31	23	20	16	10	28	21	17	14	8	23	17	14	11	6	20	14	11	9	5	1.3
1.4	27	20	17	14	9	24	18	15	12	8	20	15	12	10	6	17	12	10	8	4	1.4
1.5	24	18	15	13	8	21	16	14	11	7	18	13	11	9	5	15	11	9	7	4	1.5
1.6	21	16	14	11	7	19	14	12	10	6	16	12	10	8	5	14	10	8	6	4	1.6
1.7	19	15	13	10	7	17	13	11	9	6	14	11	9	7	4	12	9	7	6	3	1.7
1.8	17	13	11	10	6	15	12	10	8	5	13	10	8	6	4	11	8	7	5		1.8
D=δ/σ 1.9	16	12	11	9	6	14	11	9	8	5	12	9	7	6	4	10	7	6	5		1.9
2.0	14	11	10	8	6	13	10	9	7	5	11	8	7	6	4	9	7	6	4		2.0
2.1	13	10	9	8	5	12	9	8	7	5	10	8	6	5	3	8	6	5	4		2.1
2.2	12	10	8	7	5	11	9	7	6	4	9	7	6	5		8	6	5	4		2.2
2.3	11	9	8	7	5	10	8	7	6	4	9	7	6	5		7	5	5	4		2.3
2.4	11	9	8	6	5	10	8	7	6	4	8	6	5	4		7	5	4	4		2.4
2.5	10	8	7	6	4	9	7	6	5	4	8	6	5	4		6	5	4	3		2.5
3.0	8	6	6	5	4	7	6	5	4	3	6	5	4	4		5	4	3			3.0
3.5	6	5	5	4	3	6	5	4	4		5	4	4	3		4	3				3.5
4.0	6	5	4	4		5	4	4	3		4	4	3			4					4.0

Table H Values (a; b) for different (α, β): $a = \ln\frac{1-\beta}{\alpha}$; $b = \ln\frac{1-\alpha}{\beta}$

α =		**0.0005**	**0.001**	**0.005**	**0.01**	**0.02**	**0.025**	**0.04**	**0.05**	**0.10**	**0.20**	**0.25**	**0.40**	**0.50**
		7.600	6.907	5.298	4.605	3.911	3.688	3.218	2.995	2.302	1.609	1.386	0.916	0.693
	0.0005	7.600	7.600	7.596	7.591	7.581	7.576	7.560	7.550	7.496	7.378	7.313	7.090	6.908
		7.600	6.907	5.297	4.604	3.911	3.688	3.218	2.995	2.302	1.608	1.385	0.915	0.692
	0.001	6.907	6.907	6.903	6.898	6.888	6.882	6.867	6.856	6.802	6.685	6.620	6.397	6.215
		7.596	6.903	5.293	4.600	3.907	3.684	3.214	2.991	2.298	1.604	1.381	0.911	0.688
	0.005	5.298	5.297	5.293	5.288	5.278	5.273	5.257	5.247	5.193	5.075	5.011	4.787	4.605
		7.591	6.898	5.288	4.595	3.902	3.679	3.209	2.986	2.293	1.599	1.376	0.906	0.683
	0.01	4.605	4.604	4.600	4.595	4.585	4.580	4.564	4.554	4.500	4.382	4.317	4.094	3.912
		7.581	6.888	5.278	4.585	3.892	3.669	3.199	2.976	2.282	1.589	1.366	0.896	0.673
	0.02	3.911	3.911	3.907	3.902	3.892	3.887	3.871	3.861	3.807	3.680	3.624	3.401	3.219
		7.576	6.882	5.273	4.580	3.887	3.664	3.194	2.970	2.277	1.584	1.361	0.891	0.668
	0.025	3.688	3.688	3.684	3.679	3.669	3.664	3.648	3.638	3.583	3.466	3.401	3.178	2.996
		7.560	6.867	5.257	4.564	3.871	3.648	3.178	2.955	2.262	1.569	1.345	0.875	0.652
β =	0.04	3.218	3.218	3.214	3.209	3.199	3.194	3.178	3.168	3.114	2.996	2.931	2.708	2.526
		7.550	6.856	5.247	4.554	3.861	3.638	3.168	2.944	2.251	1.558	1.335	0.865	0.642
	0.05	2.995	2.995	2.991	2.986	2.976	2.970	2.955	2.944	2.890	2.773	2.708	2.485	2.303
		7.496	6.802	5.193	4.500	3.807	3.583	3.114	2.890	2.197	1.504	1.281	0.811	0.588
	0.10	2.302	2.302	2.298	2.293	2.282	2.277	2.262	2.251	2.197	2.079	2.015	1.792	1.609
		7.378	6.685	5.075	4.382	3.689	3.466	2.996	2.773	2.079	1.386	1.163	0.693	0.470
	0.20	1.609	1.608	1.604	1.599	1.589	1.584	1.569	1.558	1.504	1.386	1.322	1.099	0.916
		7.313	6.620	5.011	4.317	3.624	3.401	2.931	2.708	2.015	1.322	1.099	0.629	0.405
	0.25	1.386	1.385	1.381	1.376	1.366	1.361	1.345	1.335	1.281	1.163	1.099	0.875	0.693
		7.090	6.397	4.787	4.094	3.401	3.178	2.708	2.485	1.792	1.099	0.875	0.405	0.182
	0.40	0.916	0.915	0.911	0.906	0.896	0.891	0.875	0.865	0.811	0.693	0.629	0.405	0.223
		6.908	6.215	4.605	3.912	3.219	2.996	2.526	2.303	1.609	0.916	0.693	0.223	0.000
	0.50	0.693	0.692	0.688	0.683	0.673	0.668	0.652	0.642	0.588	0.470	0.405	0.182	0.000

DroopBooks

Table I Values of cochran's criterion for α=0.05

f_1		1	2	3	4	5	6	7	8	9	10	16	36	144	
	2	0.9985	0.9750	0.9392	0.9057	0.8584	0.8534	0.8332	0.8159	0.8010	0.7880	0.7341	0.6602	0.5813	0.5000
	3	0.9669	0.8709	0.7977	0.7457	0.7071	0.6771	0.6530	0.6333	0.6167	0.6025	0.5466	0.4748	0.4031	0.3333
	4	0.9065	0.7679	0.6841	0.6287	0.5895	0.5598	0.5365	0.5175	0.5017	0.4884	0.4366	0.3720	0.3093	0.2500
	5	0.8412	0.6838	0.5981	0.5440	0.5063	0.4783	0.4564	0.4387	0.4241	0.4118	0.3645	0.3066	0.2513	0.2000
	6	0.7808	0.6161	0.5231	0.4803	0.4447	0.4184	0.3980	0.3817	0.3682	0.3568	0.3135	0.2612	0.2119	0.1667
	7	0.7271	0.5612	0.4800	0.4307	0.3907	0.3726	0.3555	0.3384	0.3254	0.3154	0.2756	0.2277	0.1833	0.1429
	8	0.6798	0.5157	0.4377	0.3910	0.3595	0.3362	0.3185	0.3043	0.2926	0.2829	0.2462	0.2022	0.1616	0.1250
	9	0.6385	0.4775	0.4027	0.3584	0.3286	0.3067	0.2901	0.2768	0.2659	0.2568	0.2226	0.1820	0.1446	0.1111
	10	0.6020	0.4450	0.3733	0.3311	0.3029	0.2823	0.2666	0.2541	0.2439	0.2353	0.2032	0.1655	0.1308	0.1000
f_2															
	12	0.5410	0.3924	0.3264	0.2880	0.2624	0.2439	0.2299	0.2187	0.2098	0.2020	0.1737	0.1403	0.1100	0.0833
	15	0.4709	0.3346	0.2758	0.2419	0.2195	0.2034	0.1911	0.1815	0.1736	0.1671	0.1429	0.1144	0.0889	0.0677
	20	0.3894	0.2705	0.2205	0.1921	0.1735	0.1602	0.1501	0.1422	0.1357	0.1303	0.1108	0.0879	0.0675	0.0500
	24	0.3434	0.2354	0.1907	0.1656	0.1493	0.1374	0.1286	0.1216	0.1160	0.1113	0.0942	0.0743	0.0567	0.0417
	30	0.2929	0.1980	0.1593	0.1377	0.1237	0.1137	0.1061	0.1002	0.0958	0.0921	0.0771	0.0604	0.0457	0.0333
	40	0.2370	0.1576	0.1259	0.1082	0.0968	0.0887	0.0827	0.0780	0.0745	0.0713	0.0595	0.0462	0.0347	0.02250
	60	0.1737	0.1131	0.0895	0.0766	0.0682	0.0623	0.0583	0.0552	0.0520	0.0497	0.0411	0.0316	0.0234	0.0167
	120	0.0998	0.0632	0.0495	0.0419	0.0371	0.0337	0.0312	0.0292	0.0279	0.0266	0.0218	0.0165	0.0120	0.0083
	∞	0000	0000	0000	0000	0000	0000	0000	0000	0000	0000	0000	0000	0000	0000

Table J Laplace function

z	$\Phi_0(z)$									
	0	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
0.0	0.0000	0.0040	0.0080	0.0120	0.0160	0.0199	0.0239	0.0279	0.0319	0.0359
0.1	0.0398	0.0438	0.0478	0.0517	0.0557	0.0596	0.0636	0.0675	0.0714	0.0753
0.2	0.0793	0.0832	0.0871	0.0910	0.0948	0.0987	0.0026	0.0064	0.0103	0.0141
0.3	0.1179	0.1217	0.1255	0.1293	0.1331	0.1368	0.1406	0.1443	0.1480	0.1517
0.4	0.1554	0.1591	0.1628	0.1664	0.1700	0.1736	0.1772	0.1808	0.1844	0.1879
0.5	0.1915	0.1950	0.1985	0.1019	0.1054	0.1088	0.1123	0.1157	0.1190	0.1224
0.6	0.2257	0.2291	0.2324	0.2357	0.2389	0.2422	0.2454	0.2486	0.2517	0.2549
0.7	0.2580	0.2611	0.2642	0.2673	0.2703	0.2734	0.2764	0.2794	0.2823	0.2852
0.8	0.2881	0.2910	0.2939	0.2967	0.2995	0.2023	0.2051	0.2078	0.2106	0.2133
0.9	0.3159	0.3186	0.3212	0.3238	0.3264	0.3289	0.3315	0.3340	0.3365	0.3389
1.0	0.3413	0.3437	0.3461	0.3485	0.3508	0.3583	0.3554	0.3577	0.3599	0.3621
1.1	0.3643	0.3665	0.3686	0.3708	0.3729	0.3749	0.3770	0.3790	0.3810	0.3830
1.2	0.3849	0.3869	0.3888	0.3907	0.3925	0.3944	0.3962	0.3980	0.3997	0.3015
1.3	0.4032	0.4049	0.4066	0.4082	0.4099	0.4115	0.4131	0.4147	0.4162	0.4177
1.4	0.4192	0.4207	0.4222	0.4236	0.4251	0.4265	0.4279	0.4292	0.4306	0.4319
1.5	0.4322	0.4345	0.4357	0.4370	0.4382	0.4394	0.4406	0.4418	0.4429	0.4441
1.6	0.4452	0.4463	0.4474	0.4484	0.4495	0.4505	0.4515	0.4525	0.4535	0.4545
1.7	0.4554	0.4564	0.4573	0.4582	0.4591	0.4599	0.4608	0.4616	0.4625	0.4633
1.8	0.4641	0.4649	0.4656	0.4664	0.4671	0.4678	0.4686	0.4693	0.4699	0.4706
1.9	0.4713	0.4719	0.4726	0.4732	0.4738	0.4744	0.4750	0.4756	0.4761	0.4767
2.0	0.4772	0.4778	0.4783	0.4788	0.4793	0.4798	0.4803	0.4808	0.4812	0.4817
2.1	0.4821	0.4826	0.4830	0.4834	0.4838	0.4842	0.4846	0.4850	0.4853	0.4857
2.2	0.4860	0.4864	0.4867	0.4871	0.4874	0.4877	0.4880	0.4883	0.4886	0.4889
	0.4966	0.4474	0.4906	0.4263	0.4545	0.4755	0.4894	0.4962	0.4962	0.4893
2.3	0.4892	0.4895	0.4898	0.4900	0.4903	0.4906	0.4908	0.4911	0.4913	0.4915
	0.4759	0.4559	0.4296	0.4969	0.4581	0.4123	0.4625	0.4060	0.4437	0.4758
2.4	0.4918	0.4920	0.4922	0.4924	0.4926	0.4928	0.4930	0.4932	0.4934	0.4936
2.5	0.4025	0.4237	0.4397	0.4506	0.4564	0.4572	0.4531	0.4493	0.4309	0.4128
	0.4937	0.4939	0.4941	0.4942	0.4944	0.4946	0.4946	0.4949	0.4950	0.4952
	0.4903	0.4634	0.4323	0.4969	0.4574	0.4139	0.4664	0.4151	0.4600	0.4012

DropBooks

Table J (continued)

z	$\Phi_0(z)$									
	0	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
2.6	0.4953	0.4954	0.4956	0.4957	0.4958	0.4959	0.4960	0.4962	0.4963	0.4964
	0.4388	0.4729	0.4035	0.4308	0.4547	0.4754	0.4930	0.4074	0.4189	0.4274
2.7	0.4965	0.4966	0.4967	0.4968	0.4969	0.4970	0.4971	0.4971	0.4972	0.4973
	0.4330	0.4358	0.4359	0.4333	0.4280	0.4202	0.4099	0.4972	0.4821	0.4646
2.8	0.4974	0.4975	0.4975	0.4976	0.4977	0.4978	0.4978	0.4980	0.4980	0.4980
	0.4449	0.4229	0.4988	0.4726	0.4443	0.4140	0.4818	0.4476	0.4116	0.4738
2.9	0.4981	0.4981	0.4982	0.4983	0.4983	0.4984	0.4984	0.4985	0.4985	0.4986
	0.4342	0.4929	0.4498	0.4052	0.4689	0.4111	0.4618	0.4110	0.4588	0.4051
3.0	0.4986	0.4986	0.4987	0.4987	0.4988	0.4988	0.4988	0.4989	0.4989	0.4989
	0.4501	0.4938	0.4361	0.4772	0.4171	0.4558	0.4933	0.4297	0.4650	0.4992
3.1	0.4990	0.4990	0.4990	0.4991	0.4991	0.4991	0.4992	0.4992	0.4992	0.4992
	0.4324	0.4646	0.4957	0.4260	0.4553	0.4831	0.4112	0.4378	0.4636	0.4886
3.2	0.4993	0.4993	0.4993	0.4993	0.4994	0.4994	0.4994	0.4994	0.4994	0.4994
	0.4129	0.4363	0.4590	0.4810	0.4024	0.4230	0.4429	0.4623	0.4810	0.4991
3.3	0.4995	0.4995	0.4995	0.4995	0.4995	0.4995	0.4996	0.4996	0.4996	0.4996
	0.4166	0.4335	0.4499	0.4658	0.4811	0.4959	0.4103	0.4242	0.4376	0.4505
3.4	0.4996	0.4996	0.4996	0.4996	0.4997	0.4997	0.4997	0.4997	0.4997	0.4997
	0.4631	0.4752	0.4689	0.4982	0.4091	0.4197	0.4299	0.4398	0.4493	0.4585
3.5	0.4997	0.4997	0.4997	0.4997	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998
	0.4674	0.4759	0.4842	0.4922	0.4999	0.4074	0.4146	0.4215	0.4282	0.4347
3.6	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998
	0.4409	0.4469	0.4527	0.4583	0.4637	0.4689	0.4739	0.4787	0.4834	0.4879
3.7	0.4998	0.4998	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4922	0.4964	0.4004	0.4043	0.4080	0.4116	0.4150	0.4184	0.4216	0.4247
3.8	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4274	0.4305	0.4333	0.4359	0.4385	0.4409	0.4433	0.4456	0.4478	0.4499
3.9	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4519	0.4539	0.4557	0.4575	0.4593	0.4609	0.4625	0.4641	0.4655	0.4670
4.0	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4683	0.4696	0.4709	0.4721	0.4733	0.4744	0.4755	0.4765	0.4775	0.4784
4.1	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4793	0.4802	0.4811	0.4819	0.4826	0.4834	0.4841	0.4848	0.4854	0.4861
4.2	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4867	0.4872	0.4878	0.4883	0.4888	0.4893	0.4898	0.4902	0.4907	0.4911
4.3	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4915	0.4918	0.4922	0.4925	0.4929	0.4932	0.4935	0.4938	0.4941	0.4943
4.4	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4946	0.4948	0.4951	0.4953	0.4955	0.4957	0.4959	0.4961	0.4963	0.4964
4.5	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
	0.4966	0.4968	0.4969	0.4971	0.4972	0.4973	0.4974	0.4976	0.4977	0.4978
5.0	0.4999									
	0.4997									

Index

a

abstract trials 388
adequate 265
adequate linear model 314
adequate, model lackof fit 265
adequate second-order model 366
aliased/confounded effect 273
analysis of variance (ANOVA)
  one-way analysis of variance 63
  three-way analysis of variance 64
  two-way analysis of variance 63
approximation 266
arithmetic average 4
arithmetic matrix 275
auxiliary tables with several inputs 205
axial points 323

b

balanced incomplete random blocks 234
Barttlet's test for equality of variances 111
basic experiment 262, 265
basic level 185
bell-shaped 17
Bernoulli distribution 10
bias 32
biased variance 11
binomial distribution 11
B_k-designs 363
black-box model 167

c

calculated value 110
canonical analysis of the response surface 438
categorical/qualitative factor 189
center points 323
central composite orthogonal design 350
central composite rotatable design 325
central design 324
central limit theorem 34
chi-square distribution 52
classical design of experiments-one factor at a time 162
Cochran's test for equality of variances 113
coded factor 268
coefficient of determination 147
coefficient of variation 6
combined model 546
compactness 162
completely randomized block design 227
composite design 323
concordance coefficient 197
concordant domain of factor 262
conditional sum of squares 372
confidence coefficient 34
confidence intervals 31
confidence limits 34
confounded effect 273
consistency 31
constant method 236
continuous 171
continuous distributions 7
continuous D-optimality design 363
contour diagram 263
contour graph 170
contrast 80
controlcharts, upper and lower control limits 42
controllable 168
control points 490
core 323
core points 323
correlation analysis 146
crossed-design 542

d

D- and G-optimality 520
data transformation 113

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defining contrast 272
defining research problem 166
degrees of freedom 39
dependent variable (response) 120
design of experiments (DOE) 157
design matrix 268, 275
design point 82
design point of experiment-trial 185
design saturation 272
desirability function 176
determination of experimental error
  design points-trials 367
  experiment error (reproducibility variance) 367
  measurement error (determination error) 367
  replicated measurements (determinations) 367
  replicated trials 367
  trial error-variance of replicated trials 367
direct methods 234
discrete 171
discrete distributions 7
disturbance variables 168
domain of extreme experiment 190
domain of factors 190, 262
domain of response 171
D-optimal designs 520
D-optimality designs 363
Draper-Lawrence design 528

e

effect is statistically significant 110
efficiency 32
estimation theory 30
expected value
  sum of squares of the values 9
  weighted average 9
experimental center 168
experimental domain 186
experimental variance-reproducibility variance 370
experiment error 195
exponential function 121
extrapolation 265
extreme problems 169, 385
extreme vertices designs 506
extreme vertices screening designs 473

f

factor effects 169
factorial experiments with mixture 539
factor interval of variation 185
factor levels 168
factors 185
factor space 262
factor variation level 185
F-distribution 54
fictional factor 269
fractional factorial experiment 205, 267
fractional replica 268
full factorial experiment 205, 267

g

general response 173
generating ratio 172
Gibbs's triangle 481
gradient optimization methods 386
graeco-latin square 247

h

Harington's overall desirability function 176
Hartley's designs 365
Hartley's second-order designs 363
hexagonal 431
histogram 115
hypergeometrical 13
hypergeometric distribution 13

i

inadequate linear model 318
inadequate second-order model 366
incomplete random blockdesign 234
independent variables (factors) 120
interaction 82
interaction effects 271
interpolation 265
interpolation model 320
irregular replicas 268
irregular simplex 507

k

Kenworthy designs 539
Kono's designs 363

l

lack of fit 132, 267
lack of fit of regression models, lack of fit variance 377
  reproducibility variance 377
latin squares 238
α-level of significance 23
levels 185
limited 171
linear regression 121
lower levels 189

m

main effects 271
mathematical model 1, 262, 265
  deterministic 1, 2
  random 1, 2
  stochastic 1, 2
McLean and Anderson's design 512
mean average 4
mean deviation 5
mean error 372
measurement confidence 192
measurement error 191, 195
measures of variability 5
median array 4
method of least squares 123
method of random balance 203
method of steepest ascentis
  efficient 396
  inefficient 396
mixture design 465
mixture design x process factor design 542
mode, most probable value 4
model interpretation 311
multiple regression 136

n

nongradient methods of optimization 414
nonlinear regression 144
nonsymmetrical SSRD 435
normal distributions
  bell-shaped distribution 16
  Gauss' distribution 16
normal equations 140
normality of data distribution 115
norming 308
null/centerpoints 323
null point 168, 268

o

one-sided sequential testing 47
one-sided test 27
operational matrix 275
optimality 161
optimality design 307
optimization 385
optimization with multiple responses 170
orthogonal 307
orthogonal design 349
orthogonality 308
orthogonal second-order design
  (Box-Benken design) 349
outliers 118
overall desirability 181

p

partial desirability 176
partial responses 173
pentagonal 431
Pirson's criterion 116
Plackett-Burman designs 225
polynomial coefficients 169
polynomial function 121
polynomial models 169
polynomial regression 140
pooled sample variance 40
pooled variance estimates 66
population 3
power of a test 24
power of solving a replica 272
predicted-calculated response value 169
preliminary examination of subject of
  research 166
preliminary ranking of the factors 196
probability density function 14
probability model
  Bernoulli distribution 8
  single events 8
pseudocomponents 507
psychological experiment 196
pure random balance 204

q

qualitative factor 189
quantitative 171

r

random 191
randomization 161
random numbers 7
random variable
  continuous 8
  discrete random variable 8
random variations 110
range 5
rank curve 197
ranking 171
ranking of the qualitative responses 183
real factor values 268
reduced combined model 546
regression analysis 120, 160
regression coefficients 121, 169
regular replicas 268
regular simplex 415
reproducibility variance 91
research objective 166
response function 169, 262
responses 167, 170

DropBooks

response surface 169, 262
robust 191
rotatability 308
rotatable 307
rotatable design 323
Rozebum's triangle 481

s
samples
  random sample 3
  statistic sample 3
scatter diagram 147, 204
Scheffe simplex lattice design 484
second-order rotatable design
  Box-Wilson Design 323
sequential tests 46
simple general response 173
simplex centroid design 502
simplex-centroid-full factorial design 550
simplex-centroid X three process factor full factorial design 550
simplex circling 420
simplex figure 415
simplex lattice design 481
simplex lattice screening designs 469
simplex-self-directing method 415
simplex sum rotatable design 431
simplex swaying 420
singularity 171
standard deviation 6
standard error 192
standard normal distribution, standardized variable 18
starlike/axial/star 323
statistic 4
statistical analysis 367
statistical design of experiments 163
statistical estimation, intervalestimate 30
  point estimate 30
statistical hypotheses,
  alternative hypothesis 23
  primary or null hypothesis 23
  type I error 23
  type II error 23
statistical inference, hypothesis testing 22
  statistical estimation 22
statistically effective 171
statistically significant 110
statistical methods, descriptive statistics 3
  inferential statistics 3
steepest ascent (Box-Wilson's method) 388
subject of research with several responses 172
successiveness 161
symmetry 308
systematic 191
systematic variations 110

t
tabular value 110
test statistic 24
trial error 195
two-sided sequential testing 49
two-sided test 28

u
unbalanced incomplete random blocks 234
unbiased estimate of the population variance 11
uncontrollable 168
uniform distribution 14
universality 171
unlimited 171
unsaturated design 320
upper levels 189

v
variance 5
variance of group means 67

y
Yates method 276
youdens squares 252